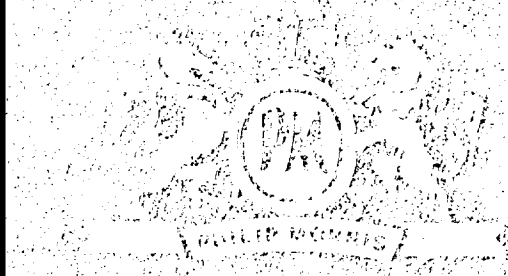


ADVISORY PROGRESS  
REPORTS



2001118136

Philip Morris U.S.A.  
Research Center  
Richmond, Virginia

Period Covered

JULY 1 - 31, 1988

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\*N/R = No Report

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PROJECT NUMBER: 2107  
PROJECT TITLE: Filter Research & Development  
PROJECT LEADER: C. J. Campbell  
PERIOD COVERED: July, 1988

I. FILTER RESEARCH & DEVELOPMENT GROUP:

- A. Objective: Consolidate efforts on filter research and development into one group and actively pursue the development of improved filter systems.
- B. Status: A new group has been formed to take on the responsibility of filter work. The initial organization of this group is complete and programs are being defined and planned.
- C. Plans: Define specific programs and establish goals.

II. CELLULOSE ACETATE WEB:

- A. Objective: Explore the potential advantages of CA web for use as a high efficiency filter material.
- B. Status: Celanese has demonstrated their capability of making plugs from CA web material supplied by Dexter. We have requested specific samples for evaluation and expect to receive these in mid August.
- C. Plans: Evaluate samples for efficiency and subjectives.

III. TOW VENDOR R&D COORDINATION:

- A. Objective: Coordinate specific requests for R&D work by Eastman and Celanese.
- B. Status: Eastman is continuing to obtain smoking data on a variety of filter configurations for Bill Dwyer's use in developing an improved smoking model. Eastman has also provided us with samples of very low denier plugs which may provide improved efficiency.

Celanese has begun some limited lab work on pre-loading tow with menthol prior to baleing. They have submitted an exclusivity agreement on this subject for our consideration.

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PROJECT NUMBER: 2108  
PROJECT TITLE: New Product Technology  
PROJECT LEADER: W. T. Callahan  
PERIOD COVERED: July, 1988

#### I. MODIFIED PLASTIC FILTER

A. Objective: Develop a filter which achieves delivery parity with Barclay.

B. Results:

Analytical data for the 50% and 60% (rear band dilution) models was received and samples have been shipped to E.E.M.A. personnel for evaluation. The FTC tar delivery was 8.1 mg (ISO tar = 9.1 mg) and 9.6 mg respectively. A right-to-use search has been requested of the Patent Group.

C. Plans: Investigate on-line and off-line methods of producing these filters at reasonable production speeds. Also, continue investigating alternate methods for flute blockage of the plastic filter.

#### II. MENTHOL ON FOIL

A. Objective: Support the introduction of menthol-on-foil process.

B. Status: Gravimetric analysis of samples from the second ethanol emission study indicated the emission level to be lower (1.24%/roll to 0.81%/roll) than that previously reported. Additional analyses are still in progress. Fabrication and calibration of the third mentholator is complete.

C. Plans: Produce samples as requested. Qualify the third mentholator for production.

#### III. PVA MENTHOL MATRIX

A. Objective: Develop a menthol system with improved menthol-in-smoke stability.

B. Status: An accelerated aging study was completed comparing menthol-on-foil, spray-applied menthol, and PVA-menthol matrix. The matrix model showed improved retention of menthol-in-smoke under lab, desert, and jungle conditions.

C. Plans: Turn over the program to the Filter R&D group (Project 2107) for further product development.

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#### IV. PROJECT HUMIDOR

- A. Objective: Develop a moisture release device for use inside a cigarette pack which maintains the pack OV at the desired level.
- B. Status: The salt solution for the production run at Klockner Packaging has been prepared at the Flavor Center. The foil laminate is being prepared by Reynolds Metals. Salt solution stability is being monitored in the lab.
- C. Plans: The foil laminate, Celgard, and salt solution are to be delivered to Klockner next week for the production run. Engineering is continuing the packer modification which is to be completed soon. QA procedures are to be developed for ensuring uniform solution quality.

#### V. EMBOSSING CONCEPTS

- A. Objective: Explore embossing technology for potential new product development.
- B. Status: PM Engineering is being provided continuing support in their development work with the lab embossing unit which has been released to them. A job order was submitted for a new lab embossing machine which will provide the capability for embossing, slitting, lamination, coating, etc. Delivery is expected by the end of September. Work has been initiated with Inta-roto exploring new embossing designs.
- C. Plans: Fabricate, install and qualify the new embossing unit.

#### VI. KAYMICH MENTHOL APPLICATOR

- A. Objective: Evaluate a Kaymich direct menthol applicator as an alternative mentholation process.
- B. Status: The Kaymich system has been installed in the Semi-works on a maker for Marlboro Lights KS and calibrated. A test plan has been prepared to compare three levels of menthol via Kaymich applicator versus menthol-on-foil.
- C. Plans: Sample manufacturing is scheduled for next week.

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PROJECT NUMBER: 2305  
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive  
PROJECT LEADER: H. M. Maxwell  
PERIOD COVERED: July, 1988

I. ALTERNATIVE FLAVORS:

- A. Objective: To identify and qualify alternative sources for flavoring materials.
- B. Status: An alternate vendor for block licorice is currently being considered. Cross-Operations mail-out panel testing is complete, and results indicate that parity was achieved. A recommendation for utilization of this material is being made. Cigarettes for POL testing of another flavor alternative (Jonex) were produced in Semi-works; they will be released for testing if they are analytically and subjectively acceptable.
- C. Plans:
- POL Testing For Alternative July, 1988  
Jonex (Merit 85)

II. PROJECT MOOG:

- A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.
- B. Status: K-type and S-type flavor optimization is in progress. Semi-works, small scale, cigarette production is scheduled for K and S flavor evaluation. A paired comparison POL testing N-type cigarettes versus Newport is scheduled to be made this week.
- C. Plans:
- K and S-Type Flavor July/August, 1988  
Development
- N-type POL (Cigarette Making) July, 1988

III. PROJECT LEVO:

- A. Objective: To determine the subjective differences between 100% natural and 100% synthetic menthol and to develop flavors to make them subjectively equivalent so that manufacturing would have complete flexibility to use them at any ratio.
- B. Status: Merit POL retesting of 100% natural vs. 100% synthetic menthol is closed out. Preliminary indications are that differences were seen. Data is being combined with that of Test I in order to better define subjective differences.

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C. Plans:

POL Results: July, 1988

Flavor Development: Ongoing

IV. MENTHOL RELEASE COMPOUND:

A. Objective: To develop a mentholated charcoal-filtered cigarette utilizing a menthol release compound.

B. Status: The Foam Bound Rod group is continuing to investigate problems of MGC application. Investigation of methods of poly isopropenyl acetate application is in progress. Development studies of the anethol/menthol release compound were initiated.

C. Plans:

Cigarette Remake (MGC): To be scheduled

V. ALTERNATE HUMECTANT PROGRAM:

A. Objective: To develop the capability to produce cigarettes that are glycerin-free and cigarettes that are propylene glycol and glycerin-free, while maintaining product subjective integrity.

B. Status: G-free POL cigarettes are being remake. Semi-works small scale G-free models, with flavor modifications, were made. Models for the PG/G-free program are being made, presently.

C. Plans:

G-free POL Remake: July, 1988

Semiworks PG/G-free Cigarette Production: July, 1988

VI. PROJECT GRAIN:

A. Objective: To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

B. Status: Equipment for flavor processing has been installed at Twentieth Street. After flavor preparation, cigarettes will be made at Stockton Street for three POL's. They will be:

1. Marlboro control versus flavor treated Marlboro;
2. Marlboro control versus Marlboro with 10% reduced alcohol; and
3. Marlboro control versus Marlboro with 30% reduced alcohol.

C. Plans:

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POL Cigarette Production

August, 1988

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PROJECT NUMBER: 2306  
PROJECT TITLE: Marlboro Standardization and International Support  
PROJECT LEADER: Janet L. Spruill  
PERIOD COVERED: July, 1988

I. MARLBORO STANDARDIZATION

- A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status: May 12 factory pick-up completed. Factory pick-ups from 6/30, 7/12 and 7/19 have been received.
- C. Plans: Analytical and subjective testing of three production dates.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. Objective: To provide subjective direction for programs within R&D and manufacturing locations.
- B. Status: Twenty panels completed during the reporting period as well as forty samples evaluated for the Menthol Aging Study and five samples for evaluations of Bright fractions.
- C. Plans: Provide assistance as needed.

III. PROJECT NATURAL

- A. Objective: To develop 85 mm full-flavored and lights prototypes using blend components and flavor systems which will provide a natural blended product.
- B. Status: Three POL's (4432-4434) have been made which will test the Virginia, Virginia/Oriental and Blended models against each other at 9 mg tar. Another POL has been requested testing Blend C using the Limited flavor system vs. Marlboro 85.
- C. Plans: Primary and make-pack run of the Limited model.

IV. FLAVOR RESOURCE DATA BANK

- A. Objective: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.
- B. Status: Training of new individual continues. Twenty-four new vendor samples are in various stages of evaluation both subjectively and analytically.
- C. Plans: Division updated with information.

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## V. INTERNATIONAL SUPPORT

### International Brands Smoking Panel

- A. Objective: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. Status: Nineteen panels completed as well as profiles of Sometime Miass 100 FTB, Camel Milds 85, and John Player Special Milds 85 from Japan.
- C. Plans: Provide assistance as needed.

### PROJECT LIGHT/ULTRA

- A. Objective: Develop cigarette models at 4, 6 and 8 mg. with a new blend and flavor system.
- B. Status: Models made at 5 and 7 mg tar using Target blend, but different casing and aftercut systems released for Danchi testing.
- C. Plans: Continue flavor development.

### PROJECT MOUNT ULTRA-JAPAN

- A. Objective: Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.
- B. Status: Three cigarette models appear to have potential for Danchi testing.
- C. Plans: Continue flavor development.

### PROJECT AVALON

- A. Objective: Development of casing and aftercut systems for a regular and mentholated product for the Asian market.
- B. Status: Currently, finished prototypes are under evaluation which are using same blend, three different casing systems, and ten aftercut systems. After model selection has been narrowed, the selected models will be reevaluated for final prototype selections.
- C. Plans: Continue subjective evaluations of models made and the development of casings and aftercuts for the program.

### NEW PRODUCT IDEAS

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- A. Objective: Develop cigarette flavors to meet demands of concept ideas for different markets.
- B. Status: Prototype selection underway for Projects Incense, Coffee and Sugar.
- C. Plans: No further development work.

#### GLOSS

- A. Objective: Develop a cigarette, for the Japanese market, using specially treated cigarette papers.
- B. Status: Second series of models made targeted at 8 mg tar using P.M. Super Lights filler, aftercut and construction but a new total blend casing incorporating two treated cigarette papers.
- C. Plans: Evaluations of second series models.

#### PROJECT STARSHIP II

- A. Objective: Produce a 12 mg cigarette for the Japanese market.
- B. Status: Two of the four models made for baseline evaluations are being remade for Danchi testing. Both models will use L&M GCC blend but different casings and aftercuts.
- C. Plans: Analytical and subjective evaluations for Danchi testing.

2001118149

PROJECT NUMBER: 2307  
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor  
Investigation/Processed Tobacco  
PROJECT LEADER: W. R. Raymond  
PERIOD COVERED: July, 1988

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Trim--Stability studies continued of six prototype A/C flavors. PG analyses were completed on Trim casings.
2. ART--Stability studies continued of two ART A/C flavor systems. Fractionation and component identification continued of extracts derived from high petroleum ether solubles ART filler. In addition to common tobacco components, a series of methyl and ethyl esters of long chain aliphatic acids has been identified. Component I.D. by GCMS has been complicated by the significant content of very high boiling components. The possibility of examining these by MS/MS is under discussion with J. Lephardt and N. Jensen.

Extraction, fractionation and analysis have begun on residues obtained from the SCF holding vessel. Initial GC profiles of hexane and 10% methanol extracts of this material while complex, appear to be considerably less so than those of filler extract fractions. Thus compositional characterization of this material may provide a more accurate idea of the nature of tobacco constituents removed by SCF extraction. Separation and identification studies will continue.

A flavor absorption study is in progress comparing anethole vapor uptake by ART versus MF Lights filler. If significant differences are noted, absorption studies may be expanded to examine other A/C flavor components.

3. Flavor Retention--Several flavor encapsulates (glassy extrudates) obtained from General Foods have been analyzed for flavor loading. Loadings meet or exceed stated G.F. values. Preliminary subjective evaluations of these materials in plug space and tobacco rod applications indicate promise for further study in cigarettes. Analytical and subjective evaluations of yeast capsules of terpeneless peppermint oil indicate similar promise. Capsule matrices of both glassy extrudate and yeast capsules appear to be subjectively neutral.
4. Miscellaneous--Flavor analyses were completed for Stockton Street Run #3 of reformulated Marlboro. A final report summarizing flavor data for all three runs was issued.

Eight compounded flavors were examined by GC and GCMS for qualification as formulation ingredients. Two were unacceptable.

GC profiles and GCMS component identifications continued with several new natural oils. GC profiles continue to be incorporated in a library file and spectra of newly identified components in a flavor MS library.

## II. PROCESSED TOBACCO:

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

1. Dry Flavor Replacement--It has been arranged with purchasing to obtain samples of native and roasted JONEX from several vendors for analytical comparison with baseline material from Chart. Subjective evaluation will follow of analytically similar samples to select additional sources.
2. ART Stem--After evaluating 3-12% citric acid on CRS from absorber trials, duplicate and modified process trials were conducted with 7.5-8.0% citric acid. Analyses of top, middle and bottom post ART DL filler and CRS showed no obvious transfer of citric acid from absorber to filler. While petroleum ether extractables (PEE) continued to concentrate on the bottom filler layer, no obvious PEE increases were observed in the absorber bed when used for single runs. However, increases were noted when the same absorber bed was used for two consecutive runs (88-167 and 88-168).
3. IS vs ES--POL tests of IS versus ES in Marlboro have been completed and are awaiting final analyses. POL testing of uncased ES from LVL production will be scheduled in August.
4. TMCI ASTA Sheet--After initial subjective evaluations, two sheet trials (10 and 22A) have been selected for large scale evaluations.

200118151



PROJECT NUMBER: 4009  
PROJECT TITLE: Smoke Studies  
PROJECT LEADER: B. L. Goodman  
PERIOD COVERED: July, 1988

#### PROJECT STUDIO

A. **Objective:** Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. **Results:**

Additional modifications to the Kimberly-Clark small particle size  $\text{CaCO}_3$  paper were tested. Two different levels and types of carboxymethyl cellulose (CMC) with and without monoammonium phosphate (MAP) were evaluated in terms of subjective response and physical characteristics. The sidestream visibility was slightly lower for the new type of CMC and higher for the samples containing MAP. The presence of MAP reduced the frequency of stains behind the charline, while it had little effect on structural holes. Analytically there were only small differences between the samples.

A model with 4.9% succinate, 0.3% low substitution CMC, and 0.5% MAP was taken to the M/C panel along with the model having 4.7% succinate on the paper. There were no significant differences in acceptability, strength, or off-taste between the two models. The model with only succinate was rated as having more added flavor by the full panel as well as the low delivery smokers. Additional work will be done with both papers at different circumferences.

Ultraslim cigarettes were made with a variety of sidestream reducing papers giving a range of visibilities. Selected models will be used for a second study of comparisons between visibility reduction numbers by light extinction and as estimated by consumers' perceptions.

A five port instrument for measuring sidestream visibility has been installed in one of the conditioned labs in the CI area. Adjustments to airflow, hood design and light scatter have been done. A computerized output is being designed by CAD. The optical parts of the new system are similar to the current single port, but utilizes a white light as the source. Power surge associated with turning on the white light arc lamp have yet to be resolved. The light beam makes a triple pass across the smokestream in a narrow hood at a distance of 17 inches above the cigarettes. The smokestream is being pulled away by a vacuum pump at a flow rate that results in the cigarettes burning at the same rate as in CI's static burn time measurements.

The new system gives good uniformity from day to day with conditions held constant, but shows large differences when the location of the cigarettes within the hood is changed. Initial visibility numbers are similar to those obtained with the single port.

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- C. Plans: Continue investigation of chemical loading on the  $\text{CaCO}_3$  paper and development of high and low level  $\text{Mg}(\text{OH})_2$  wrappers.

Start using the five port visibility equipment for data collection.

2001118153

PROJECT NUMBER: 2304, 2305, 4015  
PROJECT TITLE: New Product Development  
PROJECT LEADER: B. G. Taylor, H. Maxwell, J. B. Easley, and L. S. Wu  
PERIOD COVERED: July, 1988

I. MARLBORO/MARLBORO LIGHTS MENTHOL

- A. Objective: (1) Develop Marlboro Menthol to appeal more to Salem smokers. (2) Develop a Marlboro Lights Menthol brand extension to appeal to Salem Lights smokers.
- B. Status: National retail sales in progress since 2/1/88. Menthol monitoring continues.

POL tests #2148 (Salem Lights vs Marlboro Lights Menthol) and #2149 (Salem Lights vs Marlboro Lights Menthol with 1X menthol in filter) were completed. Marlboro Lts. Menthol achieved parity with Salem Lts. among Salem Lts. smokers. HTI testing of the same cigarettes showed no significant differences in preference, but the results were directionally in favor of Salem Lts.

Field pick-ups are being planned for menthol-in-smoke and subjective testing. PED is studying the best way to accomplish this.

C. Plans:

Monitor National Prod. Start-up  
Field Pick-ups

On-going  
Aug./Sept.

II. MARLBORO ULTRA LIGHTS

- A. Objective: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.
- B. Status: Results for POL 4424 (Merit Ultra Lights 85mm vs Half Pint 85mm) are complete. There was no significant difference between the two cigarettes for acceptability or preference ratings by the smoker groups tested (Merit Ultra Lights 85mm, Merit 85mm, Marlboro Lights 85mm, Winston Lights 85mm, or Winston Ultra Lights 85mm). Ratings on the attribute scales tested indicated that Merit 85mm smokers rated Half Pint 85mm higher on strength, off-taste, throat impact, mouthcoating, amount of smoke, amount of tobacco taste, like a full flavor cigarette, and amount of aftertaste. Merit Ultra Lights, Winston Ultra Lights, Winston Lights, and Marlboro Lights 85mm smokers did not rate the two cigarettes different for any of the attribute scales.

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Results for POL 7164 (Merit Ultra Lights 100mm vs Half Pint 100mm) indicate that Merit Ultra Lights 100mm smokers rated the Merit Ultra Lights cigarette higher on acceptability and preference; Merit 100mm smokers preferred the Half Pint 100mm cigarette; and Winston Lights 100mm smokers preferred the Merit Ultra Lights 100mm cigarette. The Merit Ultra Lights 100mm smokers rated Half Pint 100mm higher on the attribute scales of strength and off-taste; Winston Lights 100mm smokers rated the Half Pint model as faster burning.

Preliminary specifications for both the 85mm and 100mm products have been drafted.

Models to evaluate an updated LDB-2 (20% expanded) and revised LDB-2 blend (25% expanded) for puff count reduction were produced. Further work is necessary. The current Marlboro Lts. candidate is scheduled to be tested vs. Marlboro Lts. Germany (6mg.) and PMSL Germany. In addition, MUL will be tested against these products. This testing will be done among LTS smokers only.

C. Plans:

Puff Count Modifications/New Blend Evaluation  
Factory Trial/Ad/Pack Testing  
Test Market

8/88  
Sept./Oct.  
1st Qtr.'89

III. PROJECT TRIM (Ultra Slim)

- A. Objective: To develop 17 mm circumference/100 mm Ultra Slim product candidates.  
B. Status:

TRIM I:

Results from POL 7174 (Capri Regular vs. Trim I Regular) showed no significant differences in acceptability or preference. However, some attribute differences were detected. Additional flavor work has been done to reduce the harshness of Trim I. The menthol Trim I POL #9088 cigarettes are not scheduled to be made at this time due to the additional work being done on Trim I nonmenthol.

TRIM IV: (Low Sidestream)

Ad/Pack results have been released for Elan Regular and Menthol, and B&H Select Thins Regular and Menthol. The 10-047-A paper (4.7% succinate, perforated) was used for all of these products.

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Results from the Internal MC panel indicated no significant differences in acceptability, strength and off-taste between cigarettes made with 10-047-A paper and 2132-83 (4.7% succinate, .3% new CMC, and .5% MAP) paper. The 10-047-A paper was rated higher in added flavor. Due to these results, KC has been requested to make a mill run of 2132-83 paper for further POL testing.

Lowest acceptable OV and menthol equilibration study results indicate 11.3% OV is the lowest acceptable OV.

C. Plans:

Additional POL Testing	Aug./Sept.
Test Market Specifications	TBD
Factory Start-Up	TBD
Test market	TBD

IV. PROJECT ART

- A. Objective: To investigate methods to decrease the nicotine content in tobacco. To develop full-flavored, ultra low, and light products using the low nicotine tobacco products.
- B. Status: The results of the extended internal PED test of an 11mg ART vs Marlboro Lights are encouraging. The ART model was found to be not significantly different from Marlboro Lights for nontobacco taste and mouthcoating. The ART model was higher in unusual flavor and off-taste and lower in strength, satisfaction and acceptability than Marlboro Lights. However, the shape of the acceptability curve from first cigarette first pack to last cigarette third pack was not as steep as earlier ART models tested in this format.

11mg menthol extended use testing is being scheduled.

Flavor development continues on the 6, 8, 11, and 16mg tar models (menthol and non-menthol) to increase the acceptability of the extracted samples. Several casings and aftercuts have been developed and are presently being evaluated. CR chemicals are also being evaluated. Investigations are underway testing the subjectives of menthol (natural vs. synthetic vs. combination of both) and the absorption of volatile aromatic components on the ART filler.

Cigarette design development for Full Flavor, Lights and Ultra Low Tar continues. Expanded ART filler is being used to optimize the puff count on Lights and Ultra Low designs.

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C. Plans:

Product/Flavor Development  
 Internal Testing - Menthol  
 Test Market ?

On-going  
 Aug./Sept.  
 1st Qtr. '89

V. PROJECT NATURALA. Objective:

Phase I

To develop "All Natural" cigarette candidates with no additives to the tobacco.

B. Status: PMI production rejected because of spots. To be remade under different operating parameters.

POL testing of three natural blends at 8 to 9 mg tar (Flush CA Filter) to be shipped week of 8/1/88. Recessed testing to follow with same parameters.

100% blend component cigarettes to be made week of 8/1/88 to help determine cause of spotting.

KC anti-spotting papers using disodium phosphate, PVA, and inner liners on order.

C. Plans:

Spotting Tests (100% Component)	Week of 8/1/88
POL Results (8-9 Flush Filter)	9/15/88
Make Recessed POLS	8/88
Study Anti-Spotting Papers	8/88

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VI. PROJECT EXTRAA. Objective: The development of 4-8 mg product candidates that have the subjective taste of products with twice the tar.B. Status: CR chemicals continue to be subjectively evaluated. Results of the 12-month storage study of Super Juice II showed little change in the organic components at room temperature. A report will be issued.

Puff profiles of competitive market brands is completed. Will be updated as needed.

Additional casing and aftercut studies for the all lamina blend in a low density rod configuration are planned.

Work has begun on a free-standing 6mg product.

C. Plans:

Evaluate paper/ca filter variability	3rd Qtr. '88
Evaluate alternate filters	On-going
Puff profile	On-going
Low density rod program	On-going

VII. PROJECT 202

A. Objective: To develop a 2-3mg "Merit-type" cigarette.

B. Status: This is a spin-off of the Extra Project.

Three monadic POL tests were shipped in July. These tests are as follows: 1) #0158 - modified Half Pint blend with 25% expanded, Half Pint casings and AC, paper/CA dual filter, and white tipping; 2) #0159 - same as #0158 except it will have cork-on-white tipping; and 3) #0160 - PMSL blend with PMSL casings and AC.

Paper filter components from Intertaba have been received and were used for the POL tests. Paper filters from Filtrona have been requested and will be evaluated when they arrive.

Also under evaluation are an All Natural blend and a 50% expanded blend. Two new flavor systems are presently being made in Semi-works on the modified Half-Pint blend.

Awaiting receipt of PMU and PMSL from Europe. These will also be tested on the monadic Mapping Panel.

C. Plans:

Receive PMU and PMSL	8/88
Additional 202 POL Candidates	8/88
Results from POL's 0158-0160	9/18
Evaluation of Filters and Blends	On-going

VIII. PROJECT VALUE ENTRY

A. Objective: To develop products to compete in the Value Entry Categories

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**B. Status:****Alpine**

Test market in progress. HTI testing (5079/10145) of Alpine Lights vs Salem Lights were remade the week of 6/6/88 and have passed subjective screening. The Salem Lights 100's were reordered due to low FTC tar results. These cigarettes have been received and submitted for analytical smoking.

Continue blend development to replace MB4B filler with a lower cost blend.

**Target**

POL 4439 (Target Lts. vs. Cambridge Lts.) shipped 6/15/88. Scheduled close-out is 8/11/88.

POL 3592 (Target FF vs. Cambridge) O.K. subjectively, but being rechecked for FTC Tar. Possible shipping date if remake is unnecessary - week of 8/8/88.

**C. Plans:**

Complete Lts. POL	8/11/88
Ship Full Flavor (If remake unnecessary)	8/8/88
Ad/Pack Testing/Factory Trial	On-hold

**IX. B&H KING SIZE**

**A. Objective:** To develop a B&H 83mm box product with comparable subjectives of B&H 100 in both a full flavor and lights version, both nonmenthol and menthol.

**B. Status:** Baseline work has begun with the request of two nonmenthol mapping POL's which consist of B&H filler made to 85mm specs.

**C. Plans:**

Nonmenthol Mapping POL's	8/88
Blend and flavor work	On-going
Test Market	1st Qtr.'89

**X. MARLBORO BLEND REFORMULATION**

**A. Objectives:** To remove off shore tobacco from Marlboro blend.

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**B. Status:**

POL's 3584 (Phase IX with modified casings vs current production Marlboro) and 3585 (Phase IX vs current production Marlboro) shipped 7/1/88 and 7/12/88. POL's 3577 and 3578 (Phase IX's vs Winston) to be shipped week of 8/8/88. Phase IX replicates and Phase X testing to be made week of 8/1/88. Phase X will also be tested against a current production Marlboro and Winston.

**C. Plans:**

Results of Phase IX vs Marlboro	Aug.
Ship Phase IX vs Winston	8/8/88
Make Phase IX replicates and Phase X	8/1/88

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PROJECT NUMBER: 4022  
PROJECT TITLE: International Product Development  
PERIOD COVERED: July, 1988  
WRITTEN BY: A. H. Confer

I. PROJECT TOLSTOY (VARIOUS COUNTRIES)

- A. Objective: Develop an upscale Russian-style cigarette.
- B. Results: Prototypes at 10% and 20% ventilation have been requested in the 30 mm tube/19 mm filter/35 mm tobacco configuration.
- C. Plans: Due to the 12-18 month lead time required to modify equipment for producing the above configuration, a 19.5 mm tube/12 mm filter/52.5 mm tobacco configuration will be explored.

II. PROJECT DRAGON (PRC)

- A. Objective: Develop a "blended" KS cigarette to be a non-PM trademark owned and manufactured by CNTC. Tar targets: 17-19 mg. (GCF-2 factory), and less than 15 mg. (ZCF factory).
- B. Results: A book summarizing blends, casings/flavors, and cigarette construction for the proposed prototypes was prepared and presented to GCF-2 management.
- C. Plans: It was agreed that GCF-2 would make laboratory-scale samples for their subjective evaluations. Richmond will supply sample quantities of the leaf grades and casings/flavors necessary for making the prototypes.

III. PROJECT AVALON (ASIA)

- A. Objective: Define and develop product(s) to fit the Avalon marketing campaign.
- B. Results: An additional 25 prototypes were fabricated (1 blend, 3 casing systems, various aftercut flavors), and subjective screening is underway.
- C. Plans: Selected prototypes will be sent to Hong Kong in early August.

IV. PROJECT FAMOUS (VARIOUS COUNTRIES)

- A. Objective: Optimize Chesterfield as a worldwide competitor to Camel.
- B. Results: A meeting was held to review consumer test results and plan further testing.

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- C. Plans: It was agreed to test Prototype N2, modified Prototype B4, and French/Italian Chesterfield vs. market Camel in France, Switzerland, and Germany in October.

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PROJECT NUMBER: 4024  
PROJECT TITLE: Japan Product Development  
PERIOD COVERED: July, 1988  
WRITTEN BY: C. R. Lambert

I. MARLBORO

- A. Objective: To improve the subjective acceptability of the Marlboro (Full Flavor) product.
- B. Status: A Danchi consumer test evaluating the current Marlboro Japan and Marlboro Lights Japan blends with and without charcoal filters indicates a preference for charcoal filters on either blend (the current Marlboro Japan Full Flavor does not have a charcoal filter). Based upon this test along with internal evaluations, the "NEW" Marlboro Japan used in our testing will have a charcoal filter and the present Marlboro Lights Japan blend.
- C. Plans: A testing matrix (5 PMI tests and 1 Danchi) evaluating the existing and "NEW" Marlboro Japan along with Lark FF and the USA Marlboro is underway.

II. CHESTERFIELD

- A. Objective: Development of an American blended product in the 11 to 12 mg tar range.
- B. Status: Based upon internal evaluations, the L&M Milds type blend was preferred over the Famous blend when a charcoal filter was used.
- C. Plans: Two models with different casing and aftercut systems will be tested on the Danchi panel versus Camel Mild and Lucky Strike.

III. BLIND TESTING OF PRODUCT

- A. Objective: Evaluate a means other than ringtipping to remove the Mild Seven logo.
- B. Status: A consumer test has gone to Japan using cigarettes which have had their logo removed by the blasting technique. The Danchi panel returns indicate the logo removal may alter the strength of the cigarettes.
- C. Plans: A test using Marlboro (USA), Lark, the "NEW" Japanese Marlboro, and Mild Seven is scheduled, but is presently being reevaluated. All models will be treated by the "blasting" operation and will also be ringtipped.

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## IV. LIGHT/ULTRA

- A. Objective: Develop 5-7 mg. tar products to compete favorably against Mild Seven Lights.
- B. Status: A Danchi Panel is presently in Japan evaluating the 5-7 mg. products. The models include variations of blend, filters, flavors, and casings. Results should be available by early September.
- C. Plans: JTI's ultra-low entry, Frontier, will be the object of a Danchi panel test in September. Information from the Danchi presently in Japan and further internal model evaluation will determine our model selections for this Danchi.

2001118164

PROJECT NUMBER: 5001  
PROJECT TITLE: Packaging Studies  
PROJECT LEADER: H. R. Dunaway  
PERIOD COVERED: July, 1988

#### PACKAGING STUDIES

A. Objective: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. Status/Plans:

1. Residual Printing Solvents in Packaging Materials: Coordinated testing, and advised Purchasing on the acceptability of the following items for use in production:

- PMI FF 100 FT 20 and 20/200
- PMI LTS 100 FT 20 and 20/200
- Va Slims Shop Carton Insert
- Merit Taste Challenge 'dotted line' Carrier
- Va Slims Lts 120 FT 20
- B&H 100 Men SP 20
- No Frills KS SP 20/200
- Thiele-Engdahl water base lacquer DJW 5123
- Marl LS FT 20 from VFB w/PPG water lacquer
- 60mm Alpine Singapore Tipping paper
- Marl LS FT 20 CHP 3F-909D
- Marl LS FT 20 VFB 3F-925E
- PMI Lts Ctn Re-print RCS 7/15
- PM Super Lts SP 20, 20/200, Closure, and FT 6 (CWL)
- Ethyl Cellosolve sample from RCS
- PMI LTS FT 20 Re-print RCS 7/18
- Alpine KS SP 20, 20/200, Closure, and Tear Tape Singapore
- PMI solvent and ink samples (23 items) from RCS
- PMI Lts FT 20 and 20/200 resampled at Cabarrus 7/20 and 7/25
- PM Super Lts 20/200 resampled at Cabarrus 7/20
- MLS FT 20 3F-925E resampled at Cabarrus 7/20
- Va Slims 120 FT 20 resampled at Louisville 7/21
- Carton Topper Coupon with and without water lacquer
- PMI Lts 100 20/200 Solvent Decay Study sampled at 1, 3, 7, 30, & 100 hours @ 73-75° F
- PM Super Lts FT 6 resampled at S/S after weekend airing

2. Solvent Decay Rate: Upon discovering the presence of n-Propyl Cellosolve in PMI LE Lts and FF packaging materials, printed by RCS Canada, an experiment was conducted by exposing single carton blanks to a temperature of 73-75° F and sampling them at 1, 3, 7, 30, and 100 hour intervals for Headspace/GC analyses. The decay rate curve resulting from this test projected no appreciable reduction in the residual level of n-Propyl Cellosolve after 41 days of airing.

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PROJECT NUMBER: 0400  
PROJECT TITLE: Low Density Rod Development  
PROJECT LEADER: R. S. Mullins  
PERIOD COVERED: July, 1988

#### LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: To identify any problems associated with extended production runs which do not manifest themselves in typical short sample production runs, a "pseudo" production run was made on the low density cigarette maker. The maker was down for 1 hour and 29 minutes of the 5 hour and 21 minute run, giving an efficiency of 72%. The average time between stops was 7 minutes and ranged from a high of 33 minutes to a low of 10 seconds. The majority of the stops were caused by either clogging of the sideseam glue nozzle or jams at the tipper rolling block. No problems were encountered which could be attributed to binder buildup.

Sample cigarettes were produced from fillers incorporating two potential burn control additives, DAP and calcium acetate. Testing of these samples is in progress. The filler containing 4% DAP, applied before the binder, tended to produce jams under the shoe similar to those experienced previously with filler having OV's near 18%, though the OV of this filler was only 14.6%. When the filler was dried to approximately 10% OV, it ran without problems. This may indicate that the DAP acts as a humectant, softening the pectin film by increasing the film's water content.

Processing of filler for the aftercut application/tracking test and the large scale casing system evaluation has been postponed by Semiworks until early August.

Installation of the overhead transport belt system has been completed and the performance of the system is being evaluated. Two suction tape widths are being tested--a narrow one which relies upon metal guide rails to constrain the rod, and a wide one which "cups" the rod, reducing the possibility of binder or filler buildup on stationary parts. Preliminary measurements made at a maker speed of 1000 cpm using the narrow belt indicate that the temperature of the bound rod is being reduced from approximately 190°F to under 100°F in less than two feet of travel down the belt.

- C. Plans: Evaluate the performance of the overhead transport belt with both narrow and wide suction tapes. Produce low density cigarette samples for evaluation of possible casing systems and for testing of aftercut application techniques.

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PROJECT NUMBER: 1307  
PROJECT TITLE: Reconstituted Tobacco Development  
PROJECT LEADER: R. G. Uhl  
PERIOD COVERED: July, 1988

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend performance of reconstituted sheet materials.

B. Results:

1. ART Project - Subjective evaluation of ART stems in pilot RL showed no difference between 8% and 12% monopotassium citrate stems. Both are an improvement over the previous 17% monopotassium citrate ART stems, but are still subjectively detectable in 100% RL cigarettes when replacing 17% of the feedstock bright stems (only half of the projected use level). The 12% citrate stems were undetectable in 100% RL cigarettes when only 5% of the bright stems were replaced.

Marlboro blends showed no subjective differences on the large panel when either the 8% or 12% monopotassium citrate ART stems were included in pilot RL at half the projected use level (replacing 17% of the bright stems). At full utilization level (replacing 34%) the test Marlboros had lower liking scores, but this was not significant at the 95% confidence level.

Cigarettes (100% RL) representing a solubles-crossover experiment were submitted for evaluation. These would demonstrate how the subjective character attributed to ART stems is apportioned between the baseweb and the CEL.

ART stem stability studies show no significant trends in bacteria, yeast or mold counts for pre-ART citrated stems (35% OV) held in sealed containers for up to 72 hours, as-is post-ART stems (35% OV) held 24 hours, or dried post-ART stems (12% OV) held for 12 weeks.

Pilot RL sheets containing ART stems with 7.5% citric acid (included at half and full projected use levels) were made into blended cigarettes for subjective comparison to direct inclusion models.

A trial was conducted at Park 500 using coarse shredded (non-ART) stems to replace 1/3 of the feedstock bright stems (or 13% of the total feedstock). This was a necessary feasibility trial since Engineering had noted problems in pumping 100% shredded stems through an off-line Varinip extraction press. Coarse shredded stems were taken as a worst case (from a mechanical processibility standpoint) of any of the stem forms (shredded or CRS) that could be used in the ART process absorbers. There were no apparent operational problems.

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2. Humectants - POL cigarettes to evaluate a glycerin-free Marlboro blend were remade in Semiworks. This totally depleted stocks of production glycerin-free sheet materials.

PG/glycerin-free Park 500 RL and BL Plant RCB sheets, containing added isosweet as humectant, are subjectively comparable to production control sheets. Since the test sheets used scrap to replace CT (to eliminate PG and glycerin in the feedstocks), "special controls" were also produced to evaluate the effect of only the feedstock change. The "special control" RL sheets were not subjectively different, but the RCB "special control" again showed significant negative subjective qualities. This is attributed to a lack of sugars caused by replacing the large cased CT component of RCB feedstock with uncased scrap. This implies that if for any future reason CT is removed from BL Plant feedstock, sugars in the RCB flavor system should be increased. It also implies that once PG/glycerin-free CT becomes available, a PG/glycerin-free RCB can be produced using less isosweet "humectant" than required for the CT-free test sheet (sugar is the main replacement for PG/glycerin in strip casings).

Pilot RL sheets containing no humectant and PG as the only humectant have been run through Semiworks at several OV levels for survivability testing. Glycerin and isosweet sheets are scheduled for August. This test is intended to determine the efficacy of humectants vs water for reducing attrition.

C. Plans:

1. Evaluate post-ART IS treatment of 12% monopotassium citrate ART stems to enhance their subjective acceptability in RL.
2. Expedite subjective results on monopotassium citrate ART stems solubles-crossover RL sheets.
3. Expedite subjective evaluation of pilot RL sheets containing citric acid ART stems.
4. Complete Semiworks processing of individual humectant sheets.

II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. Availability of the Branson ultrasonic rental unit for Modified 150B trials has been delayed to August. Installation will be coordinated with a 2 week pilot plant outage necessary to replace fire lines serving the building sprinkler system.
2. A sample of the Takasago finished liquid flavor was received and is being evaluated by Flavor Development. Takasago is

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also sending three native extracts representing different degrees of roasting. It is proposed to utilize the Flavor Center for blending of native extracts and final sugar adjustment to specifications. The use of liquids to replace dry flavors would allow size screening on the 150B lines at Park 500 and subsequently reduce waste water treatment loadings.

3. A 3,000 lb lot of pilot RLTC containing 31% oriental scrap in the feedstock was produced at the request of Leaf Department for potential evaluation as a means of adding oriental character to test blends.

C. Plans:

1. Produce pilot RL to evaluate Chart and Takasago liquid flavors as made available from Flavor Development.
2. Complete installation of the Branson ultrasonic rental unit.

III. CIGARETTE PAPER DEVELOPMENT

A. Objective: Support development of proprietary low sidestream cigarette papers.

B. Results:

1. A dedicated handsheet lab facility is being established for the purpose of developing a proprietary position in reduced sidestream cigarette papers. A laboratory has been refurbished. Industry standard testing equipment has been received; the standard handsheet mold, flat press and dryers are scheduled for delivery in early August. A supply of flax and carbonate is on hand and technician training is in progress.
2. The University of Maine has been contracted for development work on their pilot paper machine. Maine is currently developing freeness curves to characterize our flax stock. They have been expecting delivery of the proper mesh fourdrinier wire and plan to produce control cigarette paper on their machine in late August.

C. Plans:

1. Complete the installation and debugging of handsheet laboratory equipment.
2. Review flax stock characterization data from the University of Maine and arrange the production of control sheet on their pilot machine.

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PROJECT NUMBER: 1313  
PROJECT TITLE: Semiworks Process Development  
PROJECT LEADER: W. G. Sanderson  
PERIOD COVERED: July, 1988

A. Objective: Provide engineering and technical support to improve the performance and efficiency of the Semiworks operation.

B. Results:

Kaymich Flavor Dispensing Unit (Nguyen/Osmalov) - The first Kaymich Flavor Dispensing Unit was received and installed in Semiworks make/pack on a MK9/MAXS cigarette making module configured to produce Marlboro Lights type cigarettes. After the second calibration of the unit, preliminary analytical results indicated a good correlation between the set application dosage and actual lab testing. This equipment is designed to apply menthol and/or other flavors directly to the cigarette paper during cigarette production. The application rate variation for this set-up has been quoted by the manufacturer to be no more than +/- 0.15 mg. menthol/cigarette.

Second Aftercut Flavor Application System (Nguyen) - Work continued on the second aftercut flavor application system/mixing cylinder conversion project for the Semiworks primary. Detailed drawings of the support frame work for the explosion suppression system, supplied by Rayco Industries, have been forwarded to Fenwal for approval. A purchase order has been issued to Oscar Fields for the fabrication and installation of the conveyor bypass system around the first aftercut cylinder.

Five Over Five Cartoner - Semiworks Make/Pack (Osmalov) - The five over five cartoner machine, designed by PM USA Engineering, was successfully demonstrated at Raynor & Adams machine shop and then shipped to the Semiworks make/pack. This machine is a modified boxer designed to automatically feed five test packs and five control packs into a carton for processing mail out tests. It has been set and outfitted with utilities and will be tested with make/pack personnel early next month.

C. Plans: Continue with the mixing cylinder/second aftercut flavor application system conversion. Support product development on the Kaymich Flavor Dispensing Unit. Transfer the technology developed for the TLA primary process to the Semiworks small scale primary operation.

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PROJECT NUMBER: 1333  
PROJECT TITLE: Semiworks Process Control  
PROJECT LEADER: D. A. Phan  
PERIOD COVERED: July, 1988

A. Objective: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. Results:

Hauni HT Steam Tunnel Installation (Oliver) - A new Rayco conveyor has been installed between the cutters and the Adt dryer feeder. The new conveyor has been checked out and in operation since 7/25. Installation of the steam tunnel was completed on 7/30.

Temperature Control System For Scandia Overwrappers (Phan/Sims) - Installation of a temperature control system to control the Scandia end-seal heaters for improving pack seal quality was complete. Further pack-seal testing from Osmalov indicated that the side-seal heaters also require proper temperature control. Additional controllers will be installed in August.

Second Aftercut Flavor Cylinder (Phan/Sims) - Electrical and instrumentation design for the second aftercut flavor cylinder has started. Control instruments have been ordered. Electrical installation is planned to begin the week of 9/19.

Plant Operation Support (Oliver/Sims) - Logics for the Modicon programmable controller and the touch-screen IDT system have been revised to reflect recent equipment removal and addition from the total blend silos to the Adt dryer. Electrical assistance has also been provided to in-house maintenance on the Quester steam tunnel and fluffer system removal project. Electrical drawings are being updated to reflect the above changes.

C. Plans: Complete the checkout of temperature control systems for 6 Scandia side-seal heaters. Complete electrical and instrumentation design for the second aftercut cylinder and final product reject system. Continue the installation of Onspec control software and qualification of another off-line Computrac moisture analyzer for primary. Continue providing electrical plant engineering support to the Semiworks and conduct routine QA functions.

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PROJECT NUMBER: 1503  
PROJECT TITLE: Modified Smoking Materials  
PROJECT LEADER: W. D. Winterson (Temporary)  
PERIOD COVERED: July, 1988

I. FOAMED FILLER BINDER

- A. Objective: Develop process for applying a subjectively acceptable binder to the tobacco filler during making to improve cigarette quality.
- B. Results: Analysis of historical data from this program resulted in the development of a mathematical model which effectively represents the cigarette firmness results.
- C. Plans: Prepare and issue the final report for this program.

II. LOW DENSITY ROD - BINDER APPLICATION

- A. Objective: Produce precoated filler via the batch process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.
- B. Results: Process development on the continuous coating reel continued. Additional nozzles were installed in the cylinder, bringing the total to six. Satisfactory product was produced with 3% total pectin add-on using these six nozzles to spray a 4% pectin solution. Product with 6% add-on was produced by passing the filler through the coating reel twice. Attempts to produce 6% add-on in a single pass resulted in the formation of clumps of tobacco and pectin. Work is proceeding on developing a tracer for quantifying binder application and uniformity.
- C. Plans: Continue development of the coating reel, including studies on cylinder RPM, angle, and tobacco loading. Prepare coated filler for product and low density rod maker development work. Prepare coated filler for tracer studies and for pectin coating evaluations. Reinitiate study of alternate means of applying binder.

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PROJECT NUMBER: 1806  
PROJECT TITLE: New Tobacco Processes  
SECTION LEADER: S. R. Wagoner  
PERIOD COVERED: July, 1988

I. PROJECT ART - PILOT PLANT SUPPORT

- A. Objective: To provide processes for converting and casing stem materials for the Bermuda Hundred Pilot Plant.
- B. Results: Batches of Louisville and D Pilot Plant CRS were cased with either citric acid or monopotassium citrate and shipped to the Bermuda Hundred Pilot Plant on a daily basis.

Installation of the Hauni WD tunnel into the D Pilot stem line was completed. Initial runs, checking the operation of the unit superheating IS appeared successful, and a significant filling power improvement was achieved across the unit. Further work may be required to increase the steam pressure to the tunnel to achieve optimum performance.

Testing of the Dickinson Admoist conditioning unit was completed in D Pilot Plant, and the unit was shipped to the Bermuda Hundred site. The tests successfully demonstrated the Admoist's capability to condition stems for subsequent rolling and cutting.

- C. Plans: Continue to produce stem products as required by Project ART.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. Objective: To conduct trials providing information for development of the ART commercial process.
- B. Results: In continuation of post-ART filler declumping studies, separation/steaming trials were conducted utilizing a VT separator and a modified PL separator. The results from these trials will be analyzed by Engineering so that the process design and equipment purchase for the commercial facility can proceed. The current plan is to install an air separator after the extraction process to separate any clumps that may form in the extractor vessel. The clumps will be loosened in a steam cylinder and returned to the main process flow.

A cigarette maker machinability trial was conducted at the Training Center on a Mark X maker with post-ART filler. The filler was initially processed through the Semiworks by being conditioned to 24% OV, cased, dried, and flavored with after-cut. The run appeared successful as cigarettes were made at 9000 cigarettes per minute. Samples were taken for CV/OV, sieve, and chemical analyses.

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Subjective evaluation of stems cased with monopotassium citrate continued. Direct inclusion of superheated IS (8% citrate level) replacing 5% ESB was judged to be less detectable than SHIS cased with 12% citrate. Cigarettes containing the 12% citrate stems at a 3% blend level (replacing ES) gave variable and inconclusive subjective results.

C. Plans: Continue subjective evaluations of post-ART stems.

In cooperation with the Louisville Stem Processing Facility, conduct a program to determine the factors which cause Pilot Plant CRS to produce less pressure drop at a given velocity at the BHPP than CRS from Louisville.

### III. BINDER DEVELOPMENT

A. Objective: Develop methods to produce binder systems for the foam bound rod, bonded ends, and low density rod programs.

B. Results: For the foam bonded ends program, measurements of the surface tensions of the liquid and foamed binder solution (NaCMC/licorice) were compared to the viscosity of the liquid and the rate of breakdown of the foam. The results showed a correlation between the surface tension of the liquid and the foam stability. This analysis was then conducted on various formulations of NaCMC and licorice, and showed that the concentration of gum in the solution has a much more significant effect on the foam stability than the licorice. Since all of these measurements have been made on the lab foaming system, future tests will utilize Engineering's system which has the ability to make different foam densities.

Preliminary testing of fucose as a tracer for preapplied pectin was completed. Analytical Research determined the sampling size and are satisfied with the test procedure. Preparation of the treated filler will be coordinated with Modified Smoking Materials.

C. Plans: Continue to provide support as required to the above programs.

### IV. TMCI-ASTA SHEET

A. Objective: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.

B. Results: Work to date has shown that a tobacco ground to a finer size than that currently produced at Tarragona will improve sheet quality. The current ASTA process uses tobacco dust <60 mesh. Grinding capacity trials were run in Nuway Tobacco Company using their Williams roller mill to establish the production rates for

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tobacco ground to <120 mesh, <200 mesh, and <400 mesh. The results indicate that the TSA Cadiz mill can produce the finest ground tobacco by operating more than the intended single shift to support three shifts of ASTA sheet production.

Survivability trials using a 50/50 blend of ASTA/DBC Bright, and subjective evaluation of 15% ASTA in a Spanish Marlboro blend were begun. Preliminary subjective evaluation supports higher ammonia levels than presently used in RCB.

C. Plans:

1. Complete survivability trials of Spanish ASTA products made with <60 mesh dust by the end of August.
2. Run ASTA trials in Tarragona in September to determine the tobacco grind necessary to produce a higher quality sheet. Tobacco ground to <120 mesh, <200 mesh, and <400 mesh will be evaluated.

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PROJECT NUMBER: 1810  
PROJECT TITLE: Project ART  
PROJECT LEADER: Ravi Prasad  
PERIOD COVERED: July, 1988

#### PROJECT ART

- A. Objective: To support the Commercial Design and the Flavor Development activities at the Bermuda Hundred Pilot Plant.
- B. Results: Further testing with 8% citric acid on stems confirmed improved absorption efficiency compared to control with 12% monopotassium citrate on CRS. Other advantages of citric acid include a directional improvement in filler and stem subjectives, operational ease of handling, and potential for stem usage reduction.

Preliminary results on corrosion testing of citric acid on 316 L stainless steel and inconel 600 indicate potential problems with stress corrosion cracking. Because of the test results indicating potential for erosion, a piece of metal was cut from the pilot plant stem basket for inspection. It was found to have no evidence of corrosion. However, all testing of citric acid in the pilot plant was stopped pending further evaluation of corrosion potential.

Separate clump making tests were completed, as requested by PM Engineering, to evaluate clump handling equipment for post-ART filler and stems. Generally, CRS absorber clumps more and provides higher pressure drop than the filler. However, due to the small particle size, the CRS clumps break up more readily than filler, with no need for any declumping/separating devices.

A series of runs was initiated to investigate lower AB level on DL blend filler. As expected, lower AB level results in a lowering of nicotine extraction, e.g., 88% nicotine removal @ 0% AB, 96% @ 1.6% AB, 97% @ 2.5% AB.

Pilot plant was shut down for maintenance and relief valve inspection during the week of July 11, 1988. All scheduled maintenance was completed, including the installation of new o-rings in the vessels and in the p-transmitters. The start up after maintenance activity was satisfactory, except for one relief valve malfunction. Corrective action is underway.

Two tests were completed to simulate the commercial plant CO<sub>2</sub> recovery process in order to quantify the tobacco wax level remaining on the extracted filler. Analytical results will be transmitted to the Flavor Group.

- C. Plans: Complete the tests on AB addition level in the range of 0% to 2.5% AB in order to allow the subjective evaluation of the extracted filler. Continue to support the Flavor and Product Development activity.

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PROJECT NUMBER: 1101  
PROJECT TITLE: Entomological Research  
PROJECT LEADER: D. L. Faustini  
WRITTEN BY: R. M. Lehman  
PERIOD COVERED: July, 1988

I. PHYSIOLOGICAL STUDIES OF THE CIGARETTE BEETLE (CB)

A. DISINFESTATION OF EXPORT CUT FILLER

1. Objective: To obtain ARS/APHIS approval for a Phytosanitary Certification treatment for export cut filler, export bright strip, stems and RL.
2. Results: Isolcell® laboratory studies have been modified to include tobacco for the purpose of maintaining the specified 60% R.H. (1). Ageless® laboratory studies have been completed (2).
3. Plans: Complete statistical evaluations prior to submitting data to ARS/APHIS for their evaluations in early September.

B. GLOBAL ECOLOGY

1. Objective: To determine the distribution and ecology of the CB as it relates to the processing of tobacco into cigarettes.
2. Results: The monitoring program at Universal Leaf has been expanded (3).
3. Plans: Continue monitoring this season and present a report.

C. FLAVOR EXTRACT STUDY

1. Objective: Determine the attraction of certain flavor extracts to female adult CB.
2. Results: The multichoice olfactometer, using an integrated vacuum system, has tested satisfactory for proper flow rate and direction. A study of the random behavior of mixed sex adult CBs in the olfactometer is now being conducted using no flavors/odors (control).
3. Plans: Evaluate random behavior data then evaluate male CB behavior with a known attractant (sex pheromone) (4).

D. WAREHOUSE FUMIGATIONS

1. Objective: To investigate the use of Controlled Atmospheres (CA) to disinfest bulk redried tobacco.
2. Results: CO<sub>2</sub> disinfestations were carried out in Commerce Road warehouses #44, 45, 46 and in Maury Street warehouses #9, 26, 31 and 33 from June 30 - July 8 under the guidance of Quality Assurance. This represents the final step in the current

proposal and evaluates CO<sub>2</sub> disinfestation at high ambient temperatures (5,6).

3. Plans: Prepare a final report.

**E. VISITS AND OUTSIDE PARTICIPATION**

1. A visit was made by D. L. Faustini to TACASA in Guatemala to assist in tobacco fumigation and plant inspection.
2. L. Ryan and D. L. Faustini attended and presented papers at the 18th International Congress of Entomology held in Vancouver, B.C., July 3-8, 1988.
3. A visit was made by L. Ryan to Protobaco in Colombia to assist in plant inspections.

**F. REFERENCES**

1. Drew, S. Notebook No. 7850.
2. Lehman, R. M. Notebook #8519, pp. 58-64, 79-84, 86-92.
3. L. Ryan. Memo to D. Faustini. Cigarette Beetle Pheromone Trap Monitoring Program at Universal Leaf Tobacco Company, Inc. (ULT) Facilities. July 5, 1988.
4. Minor, M. F. Notebook No. 8539, p. 45.
5. Ryan, L. Memo to D. L. Faustini. Proposal: To Investigate the Use of Controlled Atmospheres (CA) to Disinfest Bulk Redried Tobacco.
6. Lehman, R. M. Notebook No. 8519, p. 93.

2001118178

PROJECT NUMBER: 1620  
PROJECT TITLE: Electrophysiological Studies  
PROJECT LEADER: F. P. Gullotta  
WRITTEN BY: C. S. Hayes  
PERIOD COVERED: June, 1988

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. Objective: To develop methods by which to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

B. Results:

1. Cognitive NERP Study

Cognitive NERP experiments have been initiated with the goal of utilizing these responses to discriminate differences among flavorants. Employing a cognitive (odd-ball) paradigm, pairs of stimuli (natural menthol vs CO<sub>2</sub> and natural vs synthetic menthol) are being presented to subjects at different probabilities of occurrence (standard = 0.85 vs target = 0.15).<sup>1</sup> Preliminary evidence suggests that subjects are quite good at discriminating between both the menthol and CO<sub>2</sub> and the natural and synthetic menthol. That is, subjects are differentiating the targets from the standards and a late positive component is being recorded in response to the targets that is not found in the standards. Statistical analyses comparing the target and standard NERPs are currently being conducted as a means of quantifying the differences in the responses.

C. Plans: Immediate plans are to test ten subjects in the cognitive paradigm comparing natural menthol to CO<sub>2</sub> and synthetic to natural menthol. Further plans include the testing of mixtures of natural and synthetic menthol, d-menthol, (+)-isomenthol and other yet-to-be determined menthol-like compounds.

D. References:

1. Hayes, C. S. Notebook No. 8584, pp. 30-40.

2001118179

PROJECT NUMBER: 1702  
PROJECT TITLE: Optical Processing and Aerosol Research  
PROJECT LEADER: K. A. Cox  
PERIOD COVERED: July, 1988

I. INDIVIDUAL CIGARETTE INSPECTION (D. Lowitz)

- A. Objective: Develop methods for the inspection of individual cigarettes on a making machine.
- B. Results: Specifications for an acousto-optic (AO) scanning system to be used for individual cigarette inspection were determined. The calculations were carried out in collaboration with Professor A. Vanderlugt from N. C. State.
- C. Plans: Implement a high speed system for the transfer and storage of data from the CIM test table.

II. OPTICAL PACK INSPECTION, THEORETICAL (R. Maher, K. Cox)

- A. Objective: Develop and test a discriminating filter for use in an optical pack inspection system.
- B. Results: The SDF filter design algorithm was tested on a more extensive video image library. The library included 400 good images of Marlboro red soft packs, 300 with unacceptable side-to-side label registration, 400 with no closure stamps, and 400 with the closure stamp in the wrong location. All images were binary. A correlation filter was designed using 300 of the good images and was tested (numerically) on the remaining images. All images were correctly classified as acceptable or unacceptable.
- C. Plans: Test a digital filter of similar design.

III. OPTICAL PACK INSPECTION, EXPERIMENTAL (C. Harward, M. Mullins)

- A. Objective: Evaluate the Global Holonetics SMART CAMERA. Determine its effectiveness in discriminating between good and defective packs.
- B. Results: Further testing of the SMART CAMERA has been postponed until the thermally induced drift in the output can be reduced to an acceptable level.

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**IV. OPTICAL PACK INSPECTION, EXPERIMENTAL (C. Harward, M. Mullins)**

- A. **Objective:** Construct and evaluate an optical correlator-based pack inspection system.
- B. **Results:** A Vanderlugt type correlation filter was made from the image of an average good pack from the set described in II above. The filter was made using a photographic process and the system was evaluated using the other images from the set. The system was able to readily recognize side-to-side label misalignment and was able to recognize packs with the closure stamp in the wrong place with some reliability. The variation in the signal obtained from acceptable images was too large to permit reliable classification of packs with missing closure stamps. The latter limitation is expected from a Vanderlugt type filter. An SDF filter used in a similar inspection system is expected to yield superior performance.
- C. **Plans:** Evaluate a similar system employing a computer generated SDF filter.

**V. AEROSOL RESEARCH (T. Nguyen)**

- A. **Objective:** Develop a laboratory aerosol generator capable of producing highly concentrated condensation aerosols. Study the effects of both the generator parameters and the physical properties of the liquid on the concentration and particle size of the aerosol produced.
- B. **Results:** A new aerosol generating chamber was constructed and tested. The walls of the new chamber are porous, allowing more uniform cooling and mixing of the vapor as it is drawn into the chamber. As expected, the visibility of the aerosol produced by the generator was observed to be highly dependent upon the temperature and flow rate of the incoming vapor as well as the extent of dilution. Mark Alred and Woody Early (CAD) have completed the automation of the generator and integration with the light extinction instrumentation to be used to monitor the aerosols produced.

200118181

PROJECT NUMBER: 1704  
PROJECT TITLE: Supercritical Fluid Processes  
PROJECT LEADER: T. M. Howell  
PERIOD COVERED: July, 1988

I. LOW NICOTINE

- A. Objective: Bermuda Hundred Pilot Plant support.
- B. Results: DL blend was ordered to 25% OV without the addition of AB and extracted to 200 M/M at 60°C and 3800 psig. Two extractions were performed yielding 80% and 83% reduction of nicotine. The adsorber was held at 55°C in each case.
- C. Conclusions: This experiment was done to determine the pH change of the filler due to nicotine removal without AB. The pH changed from 5.2 to 4.9 after extraction. This seems to be consistent with the titration curves developed thus far.
- D. Plans: This work is ongoing.

II. LOW NICOTINE

- A. Objective: Bermuda Hundred Pilot Plant support.
- B. Results: A research plan is being developed to study the mechanism for "wax" deposition at the inlet of the filler bed during let-down. Hot hexane extracts of the filler are being prepared in an attempt to get an indication of the absolute amount of waxes that can be extracted from the filler. The collected material will then be placed in the microsystem to perform solubility studies at various temperatures and pressures. To date, wax content varies from 3.5% in unground to 6.6% in ground filler.
- C. Plans: Work is ongoing.

III. LOW NICOTINE

- A. Objective: Support for corrosion studies.
- B. Results: 18 liters of tobacco solubles were prepared for Dr. Stoner of UVA. The solutions will be used to study the corrosion effects of citric acid and mono-potassium citrate on 316 ss and on Inconel 600 in the presence of carbon dioxide.
- C. Plans: Preparation complete.

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**IV. LOW NICOTINE**

- A. **Objective:** Develop an alternate on-line nicotine monitor.
- B. **Results:** The JASCO 600 CD Spectrophotometer was received and installed on the lab micro-extraction system. Richard Davis has made several measurements of nicotine in SC-CO<sub>2</sub> (at operating conditions), and the results are very encouraging. Linearity and sensitivity indicate this method is viable as an on-line process nicotine monitor.
- C. **Plans:** This work is ongoing.

**V. LOW ALKALOID**

- A. **Objective:** Direct Supercritical Processing, Inc. in their efforts to reduce total alkaloids in tobacco using supercritical fluids.
- B. **Results:** Four experiments have been completed to date with no significant move in the level of minor alkaloids in tobacco. Experiments have been limited to CO<sub>2</sub>, no AB and OV's of 15% and 25%.
- C. **Plans:** Continue experiments extending solvent to include supercritical ethane.

200118183



PROJECT NUMBER: 1708  
PROJECT TITLE: Physical Chemistry and Process Monitoring  
PROJECT LEADER: J. L. Banyasz  
PERIOD COVERED: July, 1988

I. OPERATIONS SUPPORT (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

- A. Objective: Determine the effect of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.
- B. Results: Simulator testing has been completed on two replicate batches of a selected adhesive and its constituent base polymer in order to obtain an assessment of variability. The behavior of the three batches (original plus two replicates) of adhesive was similar with respect to viscosity, apparent density, and total solids. The corresponding base polymer batches showed a larger variability. The data are currently under evaluation to establish whether the observed differences are significant.
- C. Plans: TGA and NMR measurements will be carried out to assess the constancy of composition for these materials.

II. OPERATIONS SUPPORT (T. Van Auken and D. Gilliam, in collaboration with the Applied Technology Group)

- A. Objective: Develop a rapid method of pectin determination.
- B. Results: Samples of BL slurry as a function of cook time have been received and analyzed for several batches from the BL plant. The IA results yield a reasonable correlation to released pectin using a four-wavelength regression. The batch-to-batch variability indicates that a fairly large teaching set is required.
- C. Plans: Measurements will be continued on BL slurry.

III. OPERATIONS SUPPORT (T. Van Auken and D. Gilliam, in collaboration with the Applied Technology Group)

- A. Objective: Develop a rapid means of determining plasticizer in filter tow.
- B. Results: Two sets of standards comprised of MKS filter plugs containing triacetin over a target concentration range of 0 - 15% have been prepared and analyzed via IA. The samples are in analysis to determine the triacetin content.
- C. Plans: Measurements will be continued on filter plugs.

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IV. OPERATIONS SUPPORT (P. Henderson, in collaboration with the Applied Technology Group)

- A. Objective: Characterization of inks.
- B. Results: Test results continue to indicate formulation instability. The following instrumentation was evaluated to establish and measure ink stability and consistency: Lab Scan 2000, to determine ink color consistency; a printability tester with associated paper moisture-sensing capacity, to evaluate moisture and its effect on a standard ink application; an ink rub dryness tester, to determine the drying time of inks as a function of printing speed and ink formulation. The instruments are acceptable and will be used to develop information necessary to establish and maintain ink formulations and technologies for present and future needs. Several non-vendor sources for obtaining a consultant in the area of ink formulation are being evaluated.
- C. Plans: This work is ongoing.

V. LOW DENSITY RODS (S. Ganeriwala and S. Shelton)

- A. Objective: Compare the compression properties of low density and control rods.
- B. Results: Tensile stress-strain tests were performed on bright lamina sprayed with 2, 4, and 6% pectin solutions to various add-on levels. The 6% solution was highly viscous and difficult to apply in an even manner. In all cases, lamina stiffness increased and strain-to-break decreased with increasing add-on level. Within a given add-on level, the most dilute solution (within practical limits) gave the best results. This may be due to improved penetration with the more dilute solutions. Pectin films prepared from all three solutions were also tested. The initial concentration did not affect the results in this case. The films were at least two orders of magnitude stiffer than the lamina. The films could not be stretched to more than 1% strain because the force exceeded the 5 Kg upper limit of the dynastat.
- C. Plans: Application of hot pectin solutions will be tested to improve penetration. The effect of pectin molecular weight will be determined.

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PROJECT NUMBER: 1720  
PROJECT TITLE: Analytical Microscopy  
PROJECT LEADER: V. L. Baliga  
WRITTEN BY: K. R. Sanders  
PERIOD COVERED: July, 1988

I. LOW SIDESTREAM CIGARETTE PAPERS (Sanders)

- A. Objective: Characterize the ultrastructural changes that occur in low sidestream (LSS) papers as a function of increasing temperatures.
- B. Results: Samples of LSS paper P6SW with 35%  $\text{Mg}(\text{OH})_2$  and 5%  $\text{CaCO}_3$  were prepared as outlined by K. Maloney<sup>1</sup> which included heating samples in a controlled environment chamber to 300°C, 350°C - the dissociation temperature of  $\text{Mg}(\text{OH})_2$ , and 378°C - the temperature just prior to structural failure of the paper. These papers, plus one held at ambient conditions, were then photographed by high resolution scanning electron microscopy (HRSEM) and the photomicrographs compared for ultrastructural changes. The photomicrographs revealed no outstanding changes in either the cellulose fibers or the crystalline additives<sup>2</sup>.
- C. Conclusions: It was theorized that the crystalline additives serve as a protectant to the fiber matrix against combustion. This theory was further established by examining the surface of an ashed paper sample. These photomicrographs showed that fibers that were coated with  $\text{Mg}(\text{OH})_2$  remained intact. A cigarette which used this paper was then smoked and the ashed paper examined. Again, the  $\text{Mg}(\text{OH})_2$  seemed to protect the coated fibers. There were little effects of heating noted on those fibers or on the crystalline structure of the coating.
- D. Plans: This study showed that HRSEM photomicrograph comparisons of papers held at ambient temperatures vs combustion temperatures can be used to determine if additives serve as protectants to paper fibers and by what manner. Although the original proposal listed several papers to which this technique would be applied<sup>3</sup>, they are now no longer under consideration. This technique can be applied to other samples of interest as needed.
- E. References:
1. Maloney, K., Driscoll, D., "Temperature Dependent Air Permeation of Cigarette Paper: An Experimental Approach," P.M. Special Report #87-014, 6 February 1987.
  2. Sanders, K., "Status Report - Paper Degradation Studies as a Function of Temperature: I. Ultrastructural Characterization," Memo to V. Baliga, 8 July 1988.
  3. Baliga, V., "Paper Degradation Studies as a Function of Temperature: I. Ultrastructural Characterization," Memo to R. Cox, 27 October 1987.

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## II. ART TOBACCOS (Sanders)

- A. Objective: Determine structural and elemental changes that may occur to Bright thin and bodied, Burley thin and bodied, and Oriental tobaccos both before and after SCE.
- B. Results: Work was begun on examining the samples by HRSEM. Discussions were held with P. Echlin as to ideas for further study.
- C. Plans: Complete examination by HRSEM. Prepare samples for further analysis.
- D. References:

Sanders, K., P.M. Notebook #8612, p. 112.

## III. LOW SIDESTREAM CIGARETTE PAPERS - ANALYTICAL SUPPORT (Sanders)

- A. Objective: Examine the ultrastructure of selected cigarette papers in support of the low-sidestream project.
- B. Results: Paper samples P8FH and P8GS, both containing 35%  $\text{Mg}(\text{OH})_2$  and 5%  $\text{CaCO}_3$ , were received to determine if sample P8GS was made by the precipitation method of  $\text{Mg}(\text{OH})_2$  addition as in sample P8FH or if it was made by the gel method as in sample P6SW. All samples were examined by HRSEM. Sample P8GS was found to be more like sample P8FH than sample P6SW. The  $\text{CaCO}_3$  crystals in all of the samples were the same. However, the  $\text{Mg}(\text{OH})_2$  crystals in sample P6SW coated the flax fibers with small, rounded crystals whereas the  $\text{Mg}(\text{OH})_2$  crystals in papers P8FH and P8GS did not coat the fibers as extensively and tended to aggregate in small clusters. They also tended to be more needle-like in structure. The fibers in both P8FH and P8GS also had a looser weave than those in paper P6SW.
- C. Conclusions: Paper P8GS was made primarily by the newer precipitation method of  $\text{Mg}(\text{OH})_2$  addition unlike that used in paper P6SW.
- D. Plans: Characterize further samples on request.
- E. References:

Sanders, K., "Comparison of Papers P6SW, P8FH, and P8GS," Memo to B. Goodman, 30 June 1988.

## IV. RESPONSE TO ANALYTICAL REQUESTS (Sanders)

- A. Objective: Provide analytical support to R&D.

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**B. Results:**

One customer complaint of a mold like odor and taste noticed while smoking a Marlboro KS was investigated. The filter, paper and filler were analyzed by light microscopy (LM). No mold or other contaminants were noted<sup>1</sup>.

Another customer complaint sample of a Va. Slims Lights Menthol 100 HP was investigated for foreign material which might deliver an off odor/taste. No foreign materials were found by LM or SEM<sup>2</sup>.

Two metal screens from the Fritsch cutting mill were examined by LM to determine the size of the holes in the screens<sup>3</sup>. Photomicrographs of 12 holes from each sample plus a calibration photomicrograph were made. Sample #1 was found to have a mean hole length of 1.97 mm and width of 1.06 mm. Sample #2 mean length was 2.92 mm and width 1.88 mm. It was noted that there was a substantial amount of what looked like rust on both sides of sample #1 which had altered the size and shape of some of the holes. No rust-like material was noted on sample #2.

**C. Plans:** Characterize further samples on request.

**D. References:**

1. Sanders, K., "Analytical Microscopy Result Form," to J. Shelton, 2 June 1988.
2. Sanders, K., "Analytical Microscopy Result Form," to J. Shelton, 6 July 1988.
3. Sanders, K., "Analytical Microscopy Result Form," to B. Handy, 12 July 1988.

**V. SAFETY (Sanders)**

**A. Objective:** Provide instruction to ERT members on various aspects of first aid.

**B. Results:** Two presentations were given to ERT and first aid team members. One was given on patient assessment and the other on respiratory assessment and treatment.

**VI. MISCELLANEOUS (Sanders)**

**A. Results:** A paper has been submitted for publication to the EMSA Bulletin entitled "A Technique to Mount Specimens for SEM."

200118188

PROJECT NUMBER: 1730  
PROJECT TITLE: Plant, Cell & Tissue Culture Research  
PROJECT LEADER: I. L. Uydess  
PERIOD COVERED: July, 1988

I. TOBACCO-IDENTICAL PRESERVATIVES:

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status:

1. Phase I Preservative Screens: .

- a. A 1:1:1 mixture of C-10, C-11 and C-12 fatty acids (to a final concentration of 100 µg/ml) has been tested in the Phase I shake flask assay using B. coagulans as the target organism and 100 µg/ml each of decanoic acid and propylparaben as the controls. The fatty acid mixture was observed to have approximately the same antimicrobial (bacteriostatic) effect as the 100 µg/ml decanoic acid control, producing complete inhibition of growth over the 24 hour time course of the experiment. The 100 µg/ml paraben control, on the other hand, yielded only a marginal inhibition of growth compared to the preservative-free control.
- b. A set of shake-flask experiments was conducted to further evaluate the antimicrobial nature of decanoic acid. Lag, mid-log and late stationary phase cultures of B. coagulans were exposed to 100 µg/ml decanoic acid (in PG) in an attempt to differentiate the bactericidal vs bacteriostatic impact of this C-10 acid. Representative samples of each culture as well as a preservative-free control were plated prior to and 40 minutes after the addition of the decanoic acid to obtain viable cell (CFU) counts). The OD<sub>650</sub> of each flask was also followed over this same interval. Metabolically-active (lag) and rapidly-growing (log) phase cells were found to suffer a 0.5 - 1.5 log loss in CFUs after the addition of decanoic acid while the CFUs of non-growing, (late-) stationary cultures remained the same. This loss in viability was paralleled by decreasing OD<sub>650</sub> readings for the lag and log-phase cultures over this same interval. Thus, while the fatty acids are believed to act primarily as bacteriostatic agents (1), it would appear that decanoic acid can be cidal to a substantial portion of these bacterial populations depending upon the state and conditions of growth.
- c. Spores of several ATCC strain types and PM isolate cultures were exposed to 100 µg/ml decanoic acid in a Phase I screen in order to determine if this C-10 fatty acid was inhibitory to germination. In repeated trials, decanoic acid was observed to inhibit the outgrowth/proliferation of cells

from all of the spore types tested over a 48 hour period. Subsequent to 48 hours, however, two of the seven spore types examined (B. coagulans and PM Isolate #10A) were observed to germinate based upon an increase in the OD<sub>650</sub>. Germination was not observed in the remaining strains (including B. brevis, B. licheniformis, B. subtilis, and two unidentified SEL isolates) up to and including 96 hours of incubation.

## 2. Microbiological (Phase II Screen) Investigations:

A series of preliminary, Phase II preservative screens were initiated during this period using C-Pilot Plant SEL. The fresh SEL was first plated for initial spore and vegetative cell (CFU) counts and then dosed with various levels of decanoic acid up to and including 300 µg/ml. All flasks (+/- preservative) were then incubated at 37°C either standing or shaking @ 150 rpm in order to induce spoilage. Propylparaben in PG served as a control. CFU and pH determinations were made at 24 hour intervals over the course of the experiment. Initial observations indicated that decanoic acid @ 250 - 300 µg/ml inhibited the progression of spoilage in the C-Pilot Plant SEL under these conditions [the pH of the + decanoic acid flasks remained unchanged over the duration of the experiment compared to the rising pH of the non-shaking ("spoiling") controls]. Furthermore, several of the colony types observed in the preservative-free and paraben control flasks were found to be absent in the flasks containing 250-300 µg/ml decanoic acid.

C. Conclusions: None to be reported at this time.

D. Plans: August, 1988

1. To continue the lab-based evaluation of the C-10 thru C-12 fatty acids in Phase II screens using Pilot Plant and Park 500 SELs.
2. To initiate fermentor-based (Phase III) screens of the targeted fatty acids.

E. Reference

1. Freese, E., Sheu, C. E. and E. Galliers, "Function of Lipophilic Acids as Antimicrobial Food Additives", Nature, 241:321-325, 1973.

2001118130

PROJECT NUMBER: 1752  
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke  
PROJECT LEADER: J. O. Lephardt  
PERIOD COVERED: July, 1988

#### I. TANDEM MASS SPECTROMETER

- A. Objective: To establish a tandem mass spectrometry facility for R&D.
- B. Results: The project is in a paperwork and approvals phase. The recommendations of the committee of analytical chemists involved in the project have been formalized in both written form and oral presentation to management. The paperwork is at Finance in preparation for submission to New York management.

#### II. CURIE POINT PYROLYSIS

- A. Objective: To complete a project evaluating menthol release compounds and to train additional personnel in the use of the equipment.
- B. Results: Compounds CR-2709, CR-2710, CR-2756, CR-2757, and CR-2758 were evaluated for R. Izac by Curie Point Pyrolysis - GC/MS at both 315 and 590 C.
- C. Conclusions: Semi-quantitative data on the amount of menthol and the relative ratio of menthol to menthenes generated under pyrolysis conditions were obtained for the series of compounds. This data has been tabulated in a memo to R. Izac.
- D. Plans: M. Buckner and N. Jensen, who received training in use of the instrumentation performing this analysis, will continue work with Curie point analysis of other samples.

#### III. ASHTRAY ODOR

- A. Objective: Identification of chemical contributors to ashtray odor.
- B. Results: The odor data for the four fractions isolated by preparative gc are not yet available. The odor distribution in the third fraction, thought to be most characteristic, was first investigated by fraction collection using retention time increments of 2 minutes. The stale, earthy and roasted notes were distributed mostly in the front sections, while the smoky and sharp notes were accumulated in the later portion. Therefore, the first 2 minute window was fraction-collected and analyzed by GC/MS with odor sniffing of the gc effluents. Mass spectral identification was focused only on those peaks with odors. Twenty-nine peaks out of about 60 peaks were detected with odors such as stale, nutty, burn leaf, and green. Most of the 29 peaks

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were mixtures of at least two overlapping components, and were dominated by mass fragments characteristic of aromatic compounds - such as isopropylmethylbenzenes and ethyldimethylbenzenes - which can not explain the detected odor. Further fractionation was performed using a silica gel column with hexane, methylene chloride/methanol (9:1) and methanol as elution solvents. Separated peaks with the characteristic odor were detected in the methylene chloride/methanol fraction. Due to the dilution by the elution solvent, mass spectra obtained from the fraction were very weak and contained strong interference from the background. Therefore, to eliminate the dilution effects, chromatographic heart-cutting between two columns will be necessary to separate the interferences from the odor-bearing compounds.

C. Plans:

1. To identify the odor-bearing compounds by using dual column heart-cutting between a nonpolar and polar column coupled with odor-sniffing and mass spectral analysis.
2. To continue the collection and analysis of the rest of the fractions eluted between 9.5 and 20 minutes.

D. References:

PM Notebook 8586, pp. 62-75.

2001118192

PROJECT NUMBER: 1754  
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components  
PROJECT LEADER: J. B. Wooten  
PERIOD COVERED: July, 1988

#### SOLID STATE NMR OF TOBACCO COMPONENTS

- A. **Objective:** Determine the composition and structure of tobacco cell wall biopolymers (Jan Wooten).
- B. **Results:** The bonding pattern of the phenyl propane groups in the cell walls from several plants (wheat, a hard wood tree, and tobacco) has been investigated by hydroponically feeding the plants either [1- $^{13}\text{C}$ ], [2- $^{13}\text{C}$ ], [3- $^{13}\text{C}$ ] ferulic acid and examining the root tissue in situ by  $^{13}\text{C}$  CPMAS NMR. The ferulic acid is biosynthetically incorporated into the plant cell wall lignin which acts as a natural binder between other cell wall components. When the NMR spectra of the correspondingly labeled  $^{12}\text{C}$  plants are subtracted from the spectra of  $^{13}\text{C}$  enriched plants to remove the natural abundance  $^{13}\text{C}$  signals, the signals of the various lignin substructures originating from the ferulic acid are revealed. In CPMAS NMR, before signal integration can be used to determine the relative abundance of the different substructures, it is necessary to establish that the different carbon signals have similar polarization transfer times ( $T_{\text{CH}}$ ) and rotating frame relaxation times ( $T_{\text{CH}}^{\rho}$ ). Two representative samples were chosen for this determination, a [1- $^{13}\text{C}$ ] and a [2- $^{13}\text{C}$ ] labeled plant. A set of difference spectra was obtained for 8 cross polarization contact times for each case. The relative intensity of the signals in the [2- $^{13}\text{C}$ ] labeled sample remained constant (within 10%) for contact times between 1 and 6 ms, indicating that the  $T_{\text{CH}}$  and  $T_{\text{CH}}^{\rho}$  for each signal is similar. A significant variation of the relative intensity of the signals in the [1- $^{13}\text{C}$ ] labeled sample was observed over the same range of contact time; thus an appropriate adjustment must be made before signal integrations can be used to determine the relative abundance of the different substructures involving the [1- $^{13}\text{C}$ ] label. This latter result was anticipated because non-protonated carbons (the ferulic acid carboxyl group, C-1) generally cross polarize less efficiently than protonated carbons. The integrated signal intensities of the [1-, 2- and 3- $^{13}\text{C}$ ] labeled plants (difference spectra) were measured as well as for the synthetic DHP lignin. These data are currently being analyzed; however, the contact time variation experiment confirms that the relative signal intensities for the [1- and 2- $^{13}\text{C}$ ] labeled plants that were previously obtained with a 1 ms contact time are accurate within 10%.

2001118193

PROJECT NUMBER: 1757  
PROJECT TITLE: Analytical Flavor Specifications  
PROJECT LEADER: C. S. Kroustallis  
PERIOD COVERED: July, 1988

ANALYTICAL FLAVOR SPECIFICATIONS

A. Objective: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities.

B. Results:

All of the submitted finished specifications were recoded from AA to DM codes by Mr. Daylor and they were transmitted to Mr. E. Cook's group. Evaluations of fifteen new materials are almost finished. One of these materials showing differences was identified as being sourced from two different vendors under the same DM code. Mr. Daylor informed Mr. Cook about the differences and provisions are being made for correcting the differences.

Collaborative work with Mr. D. Watson concerning bulk materials was completed. Our aim was to produce common methodology for the evaluation of these materials. Recommended methods will be submitted by Mr. Watson to QA via Mr. Crichton for their consideration prior to implementation.

We held a mini training session with Flavor Center supervisors to familiarize them with techniques for sample preparation, analysis, and interpretation of results. Additional training will be required for technicians as soon as the supervisors think it appropriate.

C. Plans: Write finished specifications for completed materials and begin evaluations of materials from a different vendor as soon as they become available (they have already been ordered).

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PROJECT NUMBER: 1758  
PROJECT TITLE: Tobacco Cell Wall Research  
PROJECT LEADER: G. H. Bokelman  
PERIOD COVERED: July, 1988

I. SIDESTREAM REDUCTION (S. Tafur, S. Baldwin and B. Rogers)

A. Objectives: (1) Examine core vs. periphery effects of cigarette fillers to reduce the generation of sidestream smoke, (2) develop the capacity to produce both paper hand-sheets and bobbins of cigarette paper, and (3) develop a protocol for the evaluation of sidestream reduction.

B. Results: Full flavor cigarettes were core-injected with selected salts to determine if affecting the burn rate of the core of the cigarette would affect sidestream visibility. The salts included potassium citrate, potassium acetate, calcium chloride, aluminum chloride and monoammonium phosphate. Preliminary data were obtained on static burn rate and MS and SS TPM deliveries. While differences in burn rate were noted, no indication of any major effect on SS TPM was observed. The salts were added at 2, 4 and 6% add-on (pro-rated for the total weight of cigarette injected). Lower levels of potassium citrate will be tried.

The fabrication of handmade cigarettes with material loaded in the core different from periphery filler was examined. Initial attempts, made with materials of contrasting color, demonstrated that it is physically possible to achieve proper placement of the core material.

Samples of 1-2  $\mu$  particle size  $MgCO_3$  have been received from Morton Thiokol. Also a 25-lb. sample of  $CaCO_3$  in the ultra-submicron particle size range, 0.02  $\mu$ , with a surface area of 72  $m^2/g$ , has been received from Pfizer.

Forms were developed to facilitate maintaining records on the evaluation of cigarette papers and cigarette models for the sidestream reduction program.

C. Plans: Two types of handsheets will be prepared at the University of Maine (1). The first will be a double layer handsheet featuring activated carbon loading, instead of  $CaCO_3$ , for the inner wrapper and 0.02  $\mu$   $CaCO_3$  for the outer wrapper. The second type of handsheet will be similar to the first except that it will incorporate thermoplastic methyl cellulose as a retention aid.

D. Reference:

1. Baldwin, S., memo to R. Ferguson, "Preparation of Handsheets at the University of Maine," July 11, 1988.

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II. ESTIMATION OF BLEND COMPOSITION FOR CIGARETTES SOLD IN JAPAN AND CHINA  
(G. Bokelman, J. Stimler and General Analytical)

A. Objectives: As requested by Dave Rockwell, determine the Oriental tobacco content of the expanded fraction of Islands (produced by RJR Nabisco for sale in Japan). Also, predict the blend composition of Yangcheng (Filter Kings) from Guangzhou no. 2 Cigarette Factory.

B. Results: The expanded fraction of Islands, as determined by flotation in cyclopentane, represents 30.9% of the total blend. However, the Oriental leaf content of this expanded fraction is less than 2.0%. This result is comparable to that found for Winston.

The Chinese have reported that their Yangcheng (Filter Kings) contain 55% bright tobacco (including 10% expanded tobacco), 35% burley tobacco and 10% Oriental tobacco. According to Alan Confer, the Chinese apparently do utilize all of their stems, either as CRS or as a reconstituted sheet. After assuming that the stems are derived from bright and burley tobaccos, respectively, in a ratio of 55 to 35, we predict the following composition for Yangcheng: 49% bright tobacco, 39% burley tobacco and 13% Oriental tobacco. Thus it may well be that the blend information provided by the Chinese is accurate.

C. Plans: The chemical compositions of bright and burley tobaccos obtained from China, Japan and the U. S. are now being compared. Four new cigarette brands recently introduced for sale in Japan, Camel Mild (from RJR Nabisco) and Frontier Regular, Frontier Menthol and Frontier Lights (all from JTI), have been submitted for chemical analyses. As soon as these analytical values are available, we will estimate their blend compositions.

III. CHARACTERIZATION OF TOBACCO HEMICELLULOSE (S. Baldwin)

A. Objective: Characterize hemicellulose isolated from green Coker 319 tobacco.

B. Results: As part of a collaborative contract research project, Dr. Norman Lewis at VPI&SU sent us a crude sample of hemicellulose that had been extracted from green Coker 319 tobacco grown in the greenhouse at Philip Morris. Recently an additional three samples of tobacco hemicellulose were received. Based on differences among the gel permeation chromatograms of these samples and differences in their neutral sugar analyses, it can be concluded that these samples consist of differing ratios of the same, or in some cases slightly different, components. Results have not yet been obtained on methylation analyses performed on these samples.

C. Plans: The individual components within these various samples of tobacco hemicellulose will be isolated and more completely characterized.

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PROJECT NUMBER: 1902  
PROJECT TITLE: Tobacco Microbiology  
PROJECT LEADER: D. J. Ayers  
PERIOD COVERED: July, 1988

#### I. SHREDDED STEM STUDY (STORAGE OF STEMS)

- A. Objective: To determine the effect of storage on microbial counts in the pre-ART (35% OV stored for up to 24 hours) and post-ART (35% OV stored for up to 24 hours and 12% OV stored for up to 12 weeks) stems that had been sprayed with 5% citric acid.
- B. Results: In this preliminary experiment, there were no significant changes seen in bacteria counts during the pre-ART and post-ART 24 hour storage study nor were there any significant changes during the first 2 weeks of the 12 week study.
- C. Conclusions: Results from this preliminary experiment suggest that shredded stems sprayed with 5% citric acid (pre- and post-ART) can be stored for up to 24 hours with no major changes in bacterial numbers.
- D. Plans: Continue the study.
- E. References:
1. Jones, J. Notebook No. 8590, pp. 60-61.

#### II. ALTERNATE HUMECTANT PROGRAM

- A. Objective: To determine if PG/G-free RCB test sheets and available control sheets, produced in the BL Plant, differ in microbial numbers after 12 weeks of storage in the laboratory environmental rooms.
- B. Results: Bacteria, mold and yeast counts were within an expected range.
- C. Conclusions: Microbial counts did not differ significantly between the RCB control and test sheets.
- D. Plans: Document results in a memo.
- E. References:
1. Jones, J. Notebook No. 8590, p. 35, 40 and 52.

#### III. BACTOMETER CALIBRATION CURVES

- A. Objective: To generate calibration curves for determining the number of bacteria present in a sample using the Bactometer.

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- B. Results: Experiments were conducted to determine if the extraction of bacteria from tobacco samples could be done at room temperature to prevent temperature shock to the bacteria. The results indicated that there was less variation in detection times between replicate samples when the extraction was done at room temperature versus 4°C. In another series of experiments the results indicated that centrifugation of stationary phase cells had minimal impact on growth kinetics (lag time was extended for less than 10 minutes).
- C. Conclusions: Tobacco related samples can be extracted at room temperature without any obvious adverse effects on results and centrifugation at 15,000 g for ten minutes has a negligible effect on growth kinetics.
- D. Plans: Continue this study.
- E. References:
1. Chadick, D. Notebook No. 8625, p. 99.

2001118198

PROJECT NUMBER: 1904  
PROJECT TITLE: Tobacco Physiology and Biochemistry  
PROJECT LEADER: H. Y. Nakatani  
WRITTEN BY: H. Y. Nakatani and E. D. Mooz  
PERIOD COVERED: July, 1988

## I. LOW NICOTINE STUDY

- A. Objective: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.
- B. Status: Harvesting of the group 13 tobacco plants (50 plants, with an average root weight of 379 g) was completed, and the processing to the 40-65% ammonium sulfate stage of the root extracts from group 12 was done (1).

Various methods to concentrate the PMT-active samples were examined, for example, dialysis against polyvinylpyrrolidone (340,000 Daltons) or sucrose, and ammonium sulfate precipitation (65% saturation). All of the methods led to losses in PMT-activity and yield indicating the instability of the enzyme and or the fact that these methods of concentration are not suitable for our use (3,4).

Stability studies showed that propylene glycol (PG), ethylene glycol (EG) and glycerol (G) all maintained PMT-active samples at 4°C for up to 48 hr. Twenty percent EG did not maintain PMT-activity over a 24 hr period at 20°C (room temperature). The best pH condition for sample storage was found to be pH 8.0 (pH 5, 6, 7 and 8 tested) (6).

Further chromatographic studies were conducted with the phenyl-Sepharose column. Elution with 0.25 M NaCl appeared to be the most suitable condition to recover PMT activity when the column was equilibrated in the absence of EG or G. The incorporation of other washing steps (in the absence of a stabilizing agent) to precede the 0.25 M NaCl step resulted in much lower yields without significant increase in the specific activity (2,6). A 13-fold increase in specific activity of PMT over the ammonium sulfate stage was obtained by a sequential 1.5 M NaCl and 0.5 M NaCl step in the presence of 20% ethylene glycol with total recovery of PMT activity. This phenyl-Sepharose column was equilibrated in the presence of 1.5 M NaCl and 20% ethylene glycol (2,6).

Application of a sucrose concentrated sample (4-fold) to the Sephacryl gel filtration column was accomplished without decreasing the resolution of the column, i.e., the molecular weight of the peak PMT-active fraction remained the same (61,000 +/- 5000 Daltons) (4). The placement of this column as the first in the chromatographic series may alleviate down-stream processing and inherent enzyme losses.

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Further studies were conducted on characterizing PMT to insure that we are indeed isolating PMT. Five percent or less of the control PMT activities were observed for N-methylputrescine (NMP) and dipalmitoylphosphatidylethanolamine (DPPE) (5). NMP is a product of the PMT enzyme reaction and substrate for methylputrescine oxidase. DPPE is a membrane component of tobacco tissue and a physiological metabolite. Importantly, the  $K_m$  for putrescine and S-adenosylmethionine were determined to be  $8.0 \times 10^{-4}M$  and  $0.9 \times 10^{-4}M$ , respectively, similar to literature values (5). These results indicate that PMT-like activity is being isolated.

Preliminary studies in which PMT active samples have been applied to native polyacrylamide gels indicate that we can recover PMT activity which is encouraging for the identification of PMT (3).

- C. **Plans:** Extracts from the group 13 plants will be processed to the 40-65% ammonium sulfate stage. Modification of the conditions to elute PMT from phenyl-Sepharose in the presence of glycerol will be continued. Inhibitor studies will be examined for further characterizing PMT.

D. **References:**

1. Dunn, R. L. Notebook No. 7899.
2. Malik, V. Notebook No. 8542.
3. Sykes, A. Notebook No. 8526.
4. Yu, T. Notebook No. 8381.
5. Mooz, E. D. Notebook No. 8599.
6. Crockett, E. Notebook No. 8563.

II. **ALTERNATE HUMECTANTS (PG/G-FREE SHEETS/CIGARETTES) (1)**

- A. **Objective:** To produce an acceptable cigarette (domestic Marlboro-type) which is PG/G-free by the end of the third quarter of 1988.
- B. **Results:** Zero-time pad and material balance survivability tests on the production plant PG/G-free RL sheets were made in the Semi-works. Samples have been submitted for analytical evaluation. 100% test cigarettes made from production plant PG/G-free RCB and control RCB were subjectively reevaluated. The control and the PG/G-free test cigarette gave similar results, while the special control cigarette (containing no class tobacco) was considered to be atypical of RCB (2).
- C. **Plans:** Zero-time pad and material balance survivability tests on the production plant, PG/G-free RCB sheets will be made in the Semi-works in August. Small-scale, control cigarettes and

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blended, PG/G-free cigarettes (containing PG/G-free flavors and casings) will be made in the Semi-works in August for subjective and toxicological evaluations.

D. References:

1. Mooz, E. D. Notebook No. 8599.
2. Ruziak, S. PG/G-free Program: 100% RL and RCB Cigarettes Made from production Plant Sheets. Memo to E. Mooz; 1988 July 8.

III. ALTERNATE HUMECTANTS (GLYCERINE-FREE SHEET/CIGARETTES) (1)

- A. Objective: To produce an acceptable glycerine-free (G-free) cigarette (domestic Marlboro-type) for a POL test by the end of the third quarter of 1988.
- B. Results: Control and G-free (POL #3582) cigarettes were remade in the Semi-works. Samples were submitted for analytical and subjective evaluation prior to being sent out. The Third Quarter Organizational Plans for the Alternate Humectant Program for 1988 have been issued (2).
- C. Plans: Control and G-free cigarettes are scheduled to be sent out in mid-August.
- D. References:
1. Mooz, E. D. Notebook No. 8599.
  2. Mooz, Elizabeth D. Third Quarter Organizational Plans for the Alternate Humectant Program (AHP) for 1988: I. Production of a PG/G-free Cigarette and II. Production of a G-free, domestic Marlboro-type cigarette for a POL test. Memo to Dr. Robert McCuen. 1988 July 22.

2001118201

PROJECT NUMBER: 2106  
PROJECT TITLE: Cigarette Performance and Design  
PROJECT LEADER: R. W. Dwyer  
PERIOD COVERED: July, 1988

I. CIGARETTE VENTILATION LEVELS DURING SMOKING (B. Dwyer and D. Leister)

- A. Objective: Determine the effects of cigarette designs on both the filter and rod-wrapper ventilation levels as functions of puff position and puff volume.
- B. Results: A theoretical model of lit-cigarette ventilation and pressure drop has been derived and computer implemented. The model has been tested with lit-cigarette results reported by Harward. The model is in good quantitative agreement with their data.
- C. Conclusions: The greatest change in filter dilution between lit and unlit cigarettes occurs at 50% filter ventilation. This was experimentally observed by Harward. The fraction of diluent air entering the rod wrapper appears independent of filter ventilation level.
- D. Plans: This aspect of cigarette performance and design is being used to refine our expert-system delivery model.
- E. References: C. N. Harward "Simultaneous measurement of dynamic RTD, filter ventilation and carbon monoxide delivery during smoking," P.M. Special Report 85-133, 1985

II. TOBACCO ROD DELIVERIES (J. Kao)

- A. Objective: Determine the effects of puff position and puff volume on the TPM deliveries of unfiltered tobacco rods.
- B. Results: CTS has performed puff-by-puff TPM delivery analyses for us on two tobacco blends at puff volumes ranging from 15 to 45 cc. A predictive model is being developed for these data.
- C. Plans: Cigarette rods have been fabricated at a variety of rod densities and circumferences. These samples will be evaluated for TPM as a function of puff position and puff volume. Currently, the samples are being weight and circumference selected.

2001118202

PROJECT NUMBER: 2500  
PROJECT TITLE: Fundamental Chemistry  
PROJECT LEADER: J. I. Seeman  
PERIOD COVERED: July, 1988

I. FLAVOR/ODOR CHEMISTRY (Paine, Seeman)

- A. Objective: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation.
- B. Results and Plans: Two gallons of glucose menthyl carbonate in propylene glycol were delivered to Flavor Development. A large scale preparation of Di-d-neoisomenthyl 2,6-naphthalene-dicarboxylate was completed. A proposal is being developed with Professors R. Anholt and S. Simon (Duke University) to examine trigeminal effects and to develop trigeminal assays.

II. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Paine, Secor, Seeman)

- A. Objective: To obtain structural information about tobacco constituents/flavorants; to develop information on cluster formation, chemical reactions in clusters, and novel modes of controlling smoke formation.
- B. Results and Plans: A detailed literature search is nearly completed on the use of novel mineral phases as paper additives for the SS program. A variety of aromatic compounds were prepared for examination by laser spectroscopy. New approaches have been proposed to observe chemical reactions in clusters.

III. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Secor, Seeman)

- A. Objective: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.
- B. Results and Plans: Two FDA-approved ion exchangers (water insoluble, decane soluble organic acids) were examined to extract nicotine from a dilute aqueous solution. Excellent transfer was observed using Panodan-SDK, and this information is being transferred to Sepracor. HPLC analysis of the nicotine was provided by R. Izac.

IV. MISCELLANEOUS (Secor)

- A. Results and Plans: An 862 g sample of nicotine was purified by distillation, in order to meet a request for a nicotine citrate solution from Flavor Development. Together with R. Izac and J.

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Naworal, succinylated 4'-hydroxyethylnicotine was prepared, analyzed, and purified. This material will serve as the hapten for the preparation of monoclonal nicotine antibodies.

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PROJECT NUMBER: 2501  
PROJECT TITLE: Smoke Chemistry  
PROJECT LEADER: R. Comes  
PERIOD COVERED: July, 1988

## I. SIDESTREAM SMOKE

**A. Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to modification of sidestream odor and irritation; development of potential proprietary products.

**B. Status:** (1) Development continues on methods of analysis of sidestream gas phase. Materials are on order for construction of a gas phase sampling apparatus for quantitatively trapping an aliquot of sidestream gas phase. (2) Method development is underway to sample larger amounts of the semivolatile fraction of sidestream smoke so that compounds present at lower levels can be detected. (3) Visibility measurements of sidestream smoke continue on selected models. (4) Joint efforts continue on studies relating to the odor and irritation of sidestream smoke. (5) Local approval of the "650" for construction of the sidestream smoking chamber has been obtained.

**C. Results:** (1) Various adsorbents have been used in conjunction with the Tekmar desorber to attempt optimization of collection of sidestream gas phase components. These include Tenax, Carboxpack (graphitized carbon black), Carboxieve (carbon black on molecular sieve) and combinations of the above. Collection, desorption and chromatographic conditions have been altered to facilitate the analysis. (2) Ether has been investigated as a solvent for concentration of some trace materials in the semivolatile fraction. (3) A comparative study of sidestream visibility and TPM was carried out on the single port, 17-port and 5-port CORESTA smoking machines. Although, as expected, different "numbers" were obtained, the rank order of the samples was the same. A 5-port smoking machine has been modified to incorporate the best features of the single and 17-port machines and has been transferred to Development for use as the standard machine for future visibility determinations. An 8-port smoking machine has been purchased from Filtrona. (4) Joint studies continue with new samples to investigate their potential effect on sidestream odor and irritation. Methods for analytical detection and quantitation are being evaluated.

**D. Plans:** (1) Continue work on gas phase method development, analytical precision, compound identifications and comparison of gas phase from low sidestream models. (2) Better methods to concentrate volatiles in the semivolatile fraction will be investigated. A sniffing port will be built for evaluation of samples produced from this smoke fraction. (3) Conduct repetitive smokings on the modified 5-port smoking machine to insure its utility as the visibility apparatus of choice. Modify the new 8-port apparatus to test its future use as a visibility apparatus. Conduct visibility determinations as required. (4) Continue support efforts to develop

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analytical methodology to analyze sidestream smoke samples from an odor and irritation standpoint. (5) Initiate construction and instrumentation purchase upon receipt of final "650" approval for the sidestream smoking chamber.

## II. MISCELLANEOUS

- A. Final approval has been given and components have been received for construction of a new total recovery smoking machine. This machine will be used for future  $^{14}\text{C}$ -studies.
- B. A collaborative effort is underway in support of Project ART. A sample of tobacco, previously treated by the ART process, was extracted and the neutral fraction collected. Component identifications in this fraction are being conducted.
- C. Routine GC/MS and pyrolysis/GC/MS analyses are being carried out as requested.

2001118206

PROJECT NUMBER: 2525  
PROJECT TITLE: Tobacco Chemistry  
PROJECT LEADER: R. R. Izac  
PERIOD COVERED: July, 1988

#### I. NATURAL PRODUCTS CHEMISTRY

- A. Objective: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.
- B. Results: Preparative separations of a Bright tobacco subfraction using countercurrent chromatography (CCC) were continued this month. Three separations of 1g each were performed on the preparative column (2.6mm I. D., 390 mL capacity). This program is an interactive program with flavor research and Project 1758, in particular involving the isolation of solavetivone.
- C. Plans: Continue fractionation of Bright tobacco extract fractions and purification of specific compounds.
- D. References:
1. Core, M. Notebook No. 8608.

#### II. LOW NICOTINE

- A. Objective: To examine characteristics of unextracted nicotine.
- B. Results: Grafted tobacco on a tomato rootstock appears to contain only extractable nicotine. Plants are being grown to graft size. A sample of low alkaloid Burley and Bright was submitted for extraction in the Art process.
- C. Plans: Continue to investigate genetic and cultural factors which influence the amount of unextracted nicotine in tobacco materials.
- D. References:
1. Izac, R. Notebook No. 8632.
  2. Bass, R. Notebook No. 8607.
  3. West, G. Notebook No. 8559.

#### III. GREENHOUSE STUDIES

- A. Objective: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.
- B. Results: The harvest of roots from Group 12 of the hydroponic Burley 21 plants has completed. About 15.8kg of fresh root material was supplied. Group 13 has reached maturity and started to flower. These plants show good color and growth.

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A visit to the Whiteville, N.C. Tobacco Research Station was made to check on the progress of the Oriental plants (Samsun). These plants are doing very well thanks to 12 inches of rainfall. This study is for the production of U.S. grown Oriental tobacco under specified cultural practices.

The greenhouse maintenance operations including seeding, transplanting, nutrient solution preparation and other cultural tasks were completed.

C. Plans: Maintain production of fresh root tissue by hydroponic culture. Monitor the field plots as needed.

D. References:

1. Bass, R. Notebook No. 8607.
2. West, G. Notebook No. 8559.

#### IV. SUPPORT ACTIVITIES

A. Objective: To provide requested assistance for special projects.

B. Results: 1) Another 30g of  $\beta$ -cyclocitrylideneacetic acid (CR 2085) as well as 29g of  $\alpha$ -cyclocitrylideneacetic acid (CR 2256) was synthesized. 2) The purification of a nicotine derivative, succinyl derivative of 4'-( $\beta$ -hydroxyethyl)-nicotine, was accomplished for H. Secor. 3) A radio-TLC analysis of extracts obtained from the  $^{14}\text{CO}_2$ -chamber grown tobacco was completed. Results indicate a similarity to extracts obtained from greenhouse grown tobacco. 4) Twelve central pith wood core cigarettes were hand made for smoking studies involving sidestream control. The cigarettes contained 2R1 filler with a 4mm x 100mm elder wood pith core. 5) The Flue-Cured Tobacco Cooperative Variety Evaluation Farm Test and Regional Small Plot Test inspection tours in Florida, Ga., S. C., and N. C. have been completed. The 1988 test includes the controls NC95 and NC2326 and seven experimental entries.

C. Plans: Continue production of CR 2085 (200g required).

D. References:

1. Izac, R. Notebook No. 8632.
2. Bass, R. Notebook No. 8607.

2001118208

PROJECT NUMBER: 6502  
PROJECT TITLE: Environmental Tobacco Smoke  
PROJECT LEADER: C. E. Thomas  
PERIOD COVERED: July 1988

## I. QUANTITATION OF BENZENE AND TOLUENE IN MS AND SS CIGARETTE SMOKE

- A. Objective: Quantify the deliveries of benzene and toluene in MS and SS tobacco smoke of the FTR C20 and C50 Cigarettes.
- B. Results: The averaged mainstream and sidestream deliveries of benzene and toluene for the C20 and C50 cigarettes (n=5) are given in the table below:

Sample Type		MS (mg/cig)	SS (mg/cig)	SS (mg/g tobacco)
C20 Cigarette (20% Ventilation)	Benzene	0.05	0.34	0.46
	Toluene	0.10	0.75	1.03
C50 Cigarette (50% Ventilation)	Benzene	0.03	0.32	0.51
	Toluene	0.05	0.72	1.15

- C. Conclusions: The differences in the filter ventilation of the two cigarettes had little effect on the SS deliveries of benzene and toluene. The change from 20% ventilation to 50% ventilation however, decreased the MS benzene and toluene deliveries by 50 percent [1]. A memo has recently been issued detailing the procedures for smoke collection and for the gas chromatographic analysis of benzene and toluene. This memo also discusses the benzene and toluene data obtained on the Mg(OH)<sub>2</sub> circumference cigarette models [2].
- D. Plans: Benzene and toluene measurements will be performed on selected reference cigarettes in support of the SS program. An HP 5880 gas chromatograph has been obtained and it will be set-up and optimized for the procedure.
- E. References:
1. H. Randolph, PM Notebook #8475, p. 157.
  2. H. Randolph, M. Parrish, "MS and SS Benzene and Toluene Deliveries for Cigarette Models With and Without Mg(OH)<sub>2</sub> Paper at 17, 20, and 24.8-mm Circumference.", Memo to R. Fenner, July 15, 1988

## II. AMBIENT MONITORING OF ENVIRONMENTAL TOBACCO SMOKE

- A. Objective: To quantify the ambient levels of CO, particulates, and nicotine in environmental spaces using portable monitoring systems.

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- B. Results: Problems with instability of the Neotronics CO sensors have been corrected by replacing the original Neotronics sensors with new sensors by Neotronics and Transducer Research. These new sensors, in a test on the Company Jet, operated normally, and recorded frequent changes in CO concentration during the flight [1].
- C. Conclusions: It is now believed that two distinct effects were the cause of anomalous data from the Neotronics sensors. First, the Neotronics sensors were old. As the sensors aged, the low relative humidity and decreased barometric pressure found in commercial aircraft could have exacerbated the marginal operation of the sensors and resulted in negative data [2].
- D. Plans: All of the Neotronics sensors will be replaced with new sensors on a periodic basis, probably every 6 to 8 months. The tests on board the company aircraft will be repeated with the addition of a second technique for measuring CO. This alternate technique may be a Miran infrared gas analyzer if the sensitivity proves to be adequate. A survey of state-of-the-art technology is being performed in the event the Miran unit can not meet the requirements [3].
- E. References:
1. G. Baker, P.M. Notebook # 8504, pp. 63-66.
  2. E. Thomas, "Status on the Investigation of Monitoring CO on Aircraft using the PASS Units.", Memo to B. Fenner, July 8, 1988.
  3. M. Parrish, P.M. Notebook #8617, p. 66.

2001118210

PROJECT NUMBER: 6505  
PROJECT TITLE: Special Investigations/Methods Development  
WRITTEN BY: D. F. Ingraham  
PERIOD COVERED: July, 1988

## I. METHODS FOR CASINGS AND AFTERCUTS

A. Objective: To develop rapid methods for the quantitation of propylene glycol, glycerin, ethanol, water, and isosweet in casings and aftercuts.

B. Results:

Sugars in bright casing appear to break down in the injection port causing a build-up of potential interferences on the Porapak® PS column. More than four hundred injections of dilute bright casing were made on a PS column to test its durability. A slight decrease in peak resolution and baseline stability was observed after about two hundred injections, but the column was returned to an acceptable performance level by heating the oven to 225°C for two hours.

Ethylene glycol (EG) was identified as an acceptable internal standard for this method. EG is automatically dispensed into standard and sample flasks using a Unipump 300. The pump dispenses EG with a relative precision of  $\pm 0.2\%$  (2-sigma) as measured by weight. EG cannot be added directly to the diluent because of a dilution effect when preparing the concentrated stock standard.

Several changes were made in the method to improve peak resolution and decrease total run time. An increase in column length (3' to 4') and a change in diluent (methanol to n-propanol) improved the resolution of water and the solvent front permitting the use of a higher initial temperature (125°C versus 85°C). The total run time was thereby reduced by three minutes. In addition, the sugars in the bright casing are sparingly soluble in n-propanol which reduces the presence of associated background interferences.

Four unknown casings (top and bright) were supplied by E. Tucker for analysis. Good agreement was observed between the measured concentrations of water, ethanol, PG, and glycerin and the calculated concentrations based on preparation. Relative standard deviations (2-sigma) for a typical bright casing were determined to be  $\pm 4\%$  for water,  $\pm 1\%$  for propylene glycol, and  $\pm 3\%$  for glycerin.

C. Plans: 1) To complete recovery studies, 2) to determine the relative standard deviation for a typical top casing, 3) to evaluate the method for aftercuts and burley spray, and 4) to present the method to the QA Methods committee on August 2nd.

200118211

## II. SPECIFICATIONS AND ANALYSIS OF INCOMING BULK LIQUIDS

- A. Objective: To evaluate the specifications and analytical testing protocol for bulk shipments of propylene glycol, glycerin, isosweet, and ethanol (SD-4).
- B. Status: Discussions with Quality Assurance personnel were completed. The information was collated onto spreadsheets and reviewed for accuracy by the respective facilities. Recommendations for the analytical testing protocol were prepared in collaboration with C. Kroustalis and M. Zimmermann.
- C. Plans: To issue the recommendations and corresponding test procedures to J. Crichton for distribution to the QA facilities. Following, a meeting will be held with personnel from all locations to discuss these recommendations.

## III. PROJECT ART

- A. Objective: Provide analytical support to project ART.

B. Results:

Support for project ART fell into two areas. Richard Davis assumed responsibility for the ART pilot facility laboratory for two weeks. Operation of the lab was essentially uneventful although some suggestions were made to improve lab operation.

The second area was the program to provide a viable online nicotine monitor. Three alternatives are under consideration exclusive of the HPLC monitor now in place in the ART pilot facility. A GC centered device is under study by Applied Automation with the goal of a vendor supported, turnkey system. The second is the determination of nicotine online by uv spectroscopy. The third is the use of circular dichroism spectroscopy (CD) as an online tool. The uv study will await the termination of the CD study and the procurement of a suitable uv spectrometer. The CD study is in progress following the purchase and installation of a Jasco J-600 CD spectrophotometer. The unit was interfaced with a supercritical microextractor fitted with a flow-through 1-cm optical cell. Initial results have been encouraging. Sources of spectral noise have been traced to thermal gradients of the supercritical CO<sub>2</sub> within the optical cell. Heat tracing of the inlet line reduced the noise to an acceptable level. A preliminary standard curve generated within the microextractor system proved linear and time scans of an initial extraction of a previously extracted DL blend resulted in calculated values which gave acceptable agreement with offline sampling and nicotine determination by GC.

- C. Conclusions: The online CD determination of nicotine shows promise as a viable technique with acceptable sensitivity and selectivity.

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D. Plans: Monitoring of Applied Automation's progress will continue with a target date of August 26, 1988 for submission of their results. The uv study will await the inhouse demonstration of a uv instrument by Chrom-A-Scope. Studies of the CD technique will continue with an actual extraction absorption run of AB sprayed DL filler in the microextractor using potassium citrate sprayed CRS. Following this will be an attempt to measure the process stream using a stopped flow technique which should result in less background noise in the CD signal.

E. References:

Davis, R. E., "ART Online Process Nicotine Monitor - Progress Report," memo to R. Cox, July 11, 1988.

IV. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIALS

- A. Objective: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.
- B. Results: A method has been written and is awaiting approval for the headspace/gas chromatographic analysis of solvents in packaging materials. Samples have been analyzed over the past month for Flavor Development. The printers are switching to solvents that we have not encountered previously. Several of these compounds have been identified by FT-IR and GC/FT-IR/MS. One compound tentatively identified as butyl cellosolve is being detected on a regular basis. The compound has been ordered so that we can positively identify and quantitate it.
- C. Plans: Compare the performance of QA's set-up to ours. QA will take over the responsibility for these analyses once they can obtain good quality, consistent results.

V. UNEXTRACTED NICOTINE

- A. Objective: To quantitate and characterize unextracted nicotine.
- B. Results:

The effect of water content on the extraction of nicotine by methanol/ammonia was investigated. Results showed that the longer the equilibration time after addition of water, the more efficient the extraction of nicotine is. Additionally, the longer the extraction time, the more nicotine that is extracted, over a 24 hour period. The effect of extraction time is less important at higher OV's (above 20%), however. On a relative basis, these effects are more significant for ART extracted tobacco (~ 0.07% nicotine) than on unextracted filler.

Samples prepared by W. Hempfling were also analyzed for nicotine in support of this project. At this point, treatment of ART

filler with hot, 1 N sodium hydroxide extracts the most nicotine, as compared to conventional extraction or pyrolysis. More experiments along this line are planned to further characterize the source of this nicotine.

## VI. RESPONSE TO ANALYTICAL REQUESTS

A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by the project personnel during the month of July included:

Several mainstream (MS)/sidestream (SS) samples submitted by H. Randolph were analyzed for glycerin content by capillary gas chromatography. Confirmation of the presence of glycerin in these samples will be made by GC/MS. The results will be reported in a memo.

Several samples of ART stems were analyzed for potassium to confirm accurate application of potassium citrate.

Three filter samples were extracted in methanol and analyzed using capillary GC to determine the amount of triethyl citrate present in the filters. (Project 2305)

Six customer complaint samples were evaluated for contamination.

Ethanol content of foil and cigarettes from the menthol on foil process was determined in order to estimate the environmental loss of ethanol. Initial results indicate little or no loss of ethanol 12 hours after foil application and 12 hours after packing cigarettes.

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PROJECT NUMBER: 6906  
PROJECT TITLE: Biological Effects of Smoke  
PROJECT LEADER: J. M. Penn  
WRITTEN BY: G. M. Nixon and J. M. Penn  
PERIOD COVERED: July, 1988

#### I. INHIBITION OF EGF ASSAY

- A. Objective: Establish the EGF assay and determine the effects of various CSCs and related products on EGF binding.
- B. Results: A second experiment was conducted to evaluate the effects of the acid, base, and neutral fractions obtained from IT 2R1 CSC and IT 100% ES CSC at a final concentration of 150 ug/ml. As in the previous experiment, each fraction reduced cell number and inhibited EGF binding. The relative rank order of both CSC samples tested for the inhibition of EGF binding were unfractionated CSC > base > acid > neutral. These results (rank order) were similar to those observed in the previous experiment.
- C. Plans: No additional experiments are planned regarding the activity of fractions obtained from the CSCs mentioned above.
- D. References:

Patskan, G. J. Notebook No. 8674, p. 93.

#### II. GLUTATHIONE DEPLETION ASSAY (GDA)

- A. Objective: To determine the effect of the reduction in cellular GSH by 2R1 CSC on Salmonella TA98 activity of the direct acting control compound, 2-nitrofluorene (2NF).
- B. Results: With a 2R1 CSC pretreatment dose of 0.50 mg/plate, the cellular GSH level was reduced and no significant increase in 2NF activity (revertants/plate) was observed in these samples. This suggests that the reduction in GSH levels by 2R1 CSC does not affect the activity of 2NF. However, the results from this experiment were compromised by the apparent CSC toxicity. Another experiment using a lower dose of 2R1 CSC (0.25 mg/plate) was successful in eliminating 2R1 CSC toxicity as well as producing a reduction in the GSH level. However, no significant difference in 2NF activity was observed as a result of the CSC-induced reduction of GSH levels. One possible explanation for this observation could be that the GSH levels returned to control levels following CSC pretreatment and before the cells were affected by 2NF.
- C. Plans: To pretreat cells with 2R1 CSC and 2NF at the same time.
- D. References:

McCoy, W. R. Notebook No. 7127, p. 188.

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### III. PROTEIN KINASE C (PKC) WHOLE CELL ASSAY

- A. Objective: To determine the optimum cell conditions, incubation time and incubation medium for treating 3T3 cells in the PKC whole cell assay.
- B. Results: Three cell conditions were tested: quiescent, high glucose quiescent, and log phase. The incubation solutions tested were standard DMEM media and whole cell buffer (WCB) which contains neither serum nor phosphate. Samples were incubated with 200 ug fresh 2R1 CSC per ml for 15 min., 5 hr., or 19.5 hr. Results indicated that cells require incubation in standard media while being exposed to CSC for extended periods of time. Log phase cells show increased levels of phosphorylation after exposure to CSC.
- C. Plans: Repeat the different incubation times of log phase cells with CSC. Test several compounds for use as positive controls. Test stored CSC.
- D. References:

Nixon, G.M. Notebook No. 8569, p. 179.

2001118216

PROJECT NUMBER: 6908  
PROJECT TITLE: Smoke Condensate Studies  
PROJECT LEADER: R. D. Kinser  
PERIOD COVERED: July, 1988

## I. ORIENTAL TOBACCO STUDIES

- A. Objective: To ascertain the existence of a chemical inhibitor to TSNA formation/pyrosynthesis in oriental tobacco.
- B. Results: Fillers were prepared by addition of burley (Bu), oriental (Ori) and mixed Bu and Ori CELs to Bu base web and were smoked to determine MS TSNA deliveries in a replicate of a previously reported experiment. MS NNN and NAT levels were again significantly reduced when Ori CEL was added to the Bu CEL.

Acid, base, and neutral fractions of Ori CEL were prepared for model studies of possible inhibition of TSNA formation by a component of Ori tobacco.

- C. Plans: Investigate the effect of Ori CEL fractions on the formation of TSNA in a model alkaloid/nitrite system.

D. References:

Haut, S. A. Notebook No. 8595, p. 114.  
Warfield, A. H. Notebook No. 8558, p. 178.

## II. TSNA DECOMPOSITION STUDIES

- A. Objective: To explore the thermal stability of TSNA and investigate methods to enhance thermally induced decompositions of these compounds.
- B. Results: Addition of ascorbyl palmitate (AP) to NNN followed by a slow heating regimen (5°C/min) shifted the temperature of onset of NNN decomposition from 220°C to 140°C and increased the extent of decomposition from 35% to 80%. No enhancement in NNN decomposition due to the addition of propyl dihydroxyhydrocinnamate was observed over the temperature range 200 - 350°C.

- C. Plans: Replicate the slow heating experiment with NNN and AP.

D. References:

Morgan, W. R. Notebook No. 8579, p. 47.  
Tickle, M. H. Notebook No. 8587, p. 189.

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### III. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Experiments to evaluate N-isobutenyl nicotinium citrate as a model of unextracted nicotine have been cancelled due to problems with synthesis of the compound.
- C. Plans: Investigate the use of microencapsulated nicotine bitartrate and a nicotine amino acid as alternate models for unextracted nicotine.
- D. References:

Haut, S. A. Notebook No. 8595, p. 114.

### IV. TSNA IN PROJECT ART MATERIALS

- A. Objective: To determine the TSNA or alkaloid levels of fillers of lowered nicotine content or other materials generated in Project ART studies.
- B. Results: A method for alkaloid analysis by extraction with benzene/ chloroform in the presence of barium hydroxide followed by quantitation of alkaloids by hplc has been developed. This method is being used to examine samples generated by extraction of tobacco with supercritical fluids.
- C. Plans: Provide analyses of fillers and other materials associated with the extraction of tobacco by supercritical fluids as requested.
- D. References:

Lambert, E. A. Notebook No. 8523, p. 168.  
Warfield, A. H. Notebook No. 8558, p. 178.

### V. OPTIMIZATION OF A LOW ACTIVITY MODEL: MS NITRIC OXIDE CONTENT

- A. Objective: To develop methods of reducing MS NO delivery for a low activity model.
- B. Results: Method development for measurement of MS NO levels continued. An interference in the first hydrogen peroxide solution utilized was shown to be the source of the high NO value obtained originally for the test cigarettes. A new lot of hydrogen peroxide, however, yielded a measurement that was significantly lower than the CI value for these cigarettes. Evaluation of a nitrate standard solution by the standard addition procedure gave good results, but similar studies of the test cigarettes yielded ambiguous information.

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C. Plans: Examine different concentrations of hydrogen peroxide and oxidation conditions. Evaluate performance of chemiluminescent detection (thermal energy analyzer) for NO measurement.

D. References:

Levins, R. J. Notebook No. 8672, p. 14.

VI. SUPPORT FUNCTION: CONDENSATE PREPARATION

A. Objective: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.

B. Results: Thirty-five fillers were prepared by spraying bright base web with burley CEL, calcium acetate, and mixtures of Bu CEL and calcium acetate, and cigarettes were hand-fabricated from these fillers. Twenty smokings of five cigarette codes were performed to collect condensate for biological assay.

C. References:

Hellams, R. D. Notebook No. 8613, p. 94.

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PROJECT NUMBER: 0008  
PROJECT TITLE: Computer Applications/ Lab Systems  
PROJECT LEADER: F. M. Allred  
PERIOD COVERED: July 1988

### I. General Analytical LIMS Migration

- A. Objective: Move General Analytical LIMS and data collection functions off the HP 1000 LABSAM and LAS system.
- B. Results: Data collection from gravimetric instruments has been off-loaded from the HP 1000. PC Lab Workstations are used to collect and manage gravimetric data. Approved results are transferred to the HP 1000 in batch mode. If the HP is not available, tests can still be run at the gravimetric instruments.

Sample Logins are now done from the new General Analytical LIMS application on the UNIX host. Automated procedures transfer the login information to the HP and update the LABSAM database.

- C. Plans: Test selected chromatography instrument with Nelson Analytical chromatography system. Compare results with current system. Use manual entry of results initially.

### II. CTSD Modernization

- A. Objective: Eliminate obsolete and difficult to maintain equipment in the CTSD area. Improve reliability of CTSD operations.
- B. Results: Collected data for a full day's run of the 4 channel Gas Phase Station.

Completed Gas Phase System Design Verification stage. Each 5-port smoker will have a Data Collection PC running MS DOS and Labtech Notebook. This PC will be coupled to an Analysis PC that will interact with the user and bar-code reader and generate reports.

- C. Plans: Develop analysis procedures and reports. Test the data collection unit with the new smoker controller being developed by Development Engineering.

### III. Tobacco Lot Analysis Data

- A. Objective: Supply Information Services with Tobacco Lot Analysis information upon request.
- B. Results: R. Lipps met with R. Rowekamp and finalized the transfer file format. M. Gillie prepared a TK50 tape with the TLA information on it and sent it to R. Rowekamp.

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#### IV. Local Area Network

A. Objective: Find a peer-to-peer local area network that works with Apple Macintosh computers, IBM Personal Computers, and computers running UNIX.

B. Results: The TOPS product from SUN was evaluated by S. Peterson and was found to have the required functionality. PCs and Macintoshes configured with TOPS can share files and printers without requiring a server. By connecting the TOPS network to a Kinetics TOPS/Ethernet gateway, and installing the appropriate software on a UNIX server, TOPS files can be shared with UNIX systems and vice versa. Printers on the TOPS network are accessible to UNIX.

A TOPS network is installed in PED. It connects two Macintosh II computers and one Club 286 (IBM PC clone). A Kinetics box allows these computers to communicate with the UNIX host.

C. Plans: Install a TOPS network in the Flavor Lab to connect four Club 286 computers.

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PROJECT NUMBER: 8101  
PROJECT TITLE: Cigarette Testing Services Division  
SECTION LEADER: Jane Y. Lewis  
PERIOD COVERED: July, 1988

## I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. American Tobacco Company

The Carlton cigarettes listed are being nationally distributed with new package graphics. These graphics incorporate a new black lettering style. No changes were noted in smoke deliveries or cigarette construction for these brands. Carlton 85 (Box) has been renamed Ultra Carlton and the package was changed from red/white to blue/white. The tar and nicotine values listed on the packs are as follows:

	<u>Tar</u>	<u>Nicotine</u>
Ultra Carlton 85 (Box)	<0.01	<0.002
Carlton 85 plain and menthol	1	0.1
Carlton 100 plain	3	0.3
Carlton 100 menthol	5	0.5
Carlton 100 (Box) plain & men.	1	0.1
Carlton 120 plain & men.	6	0.6

2. R. J. Reynolds

A change to a more porous cigarette paper (19 to 15 seconds) resulted in lower tar (10 to 9 mg), nicotine (0.7 to 0.6 mg) and CO (12 to 11 mg) deliveries for Winston Lights 85 cigarettes. A lower total RTD was also observed (143 to 119 mm of H<sub>2</sub>O). The ventilation type changed from electrostatic to laser perforation. Winston Lights 85 cigarettes continue to have cork tipping paper.

## II. METHODS DEVELOPMENT AND SUPPORT

A. Objective: To evaluate and recommend new technology in support of programs for R&D and Manufacturing.

B. Results:

1. Graphs for C.I. Report

In conjunction with CAD, software has been developed which will enable us to produce the graphs for the C.I. Report

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using the PS Laser Printer. The data for the graphs are extracted directly from the C.I. database and copied to files for production of graphs. Previously the graphs were produced manually.

2. Product Natural

At the request of personnel from Quality Engineering, the testing and calculation of cylinder volume/oven volatiles (CV/OV) laboratory equilibration factors will be established for Product Natural. Development of these empirical factors for all new tobacco blend formulations is a service which has been transferred to Cigarette Testing Services Division.

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